Flow Reactor Studies with Nanosecond Pulsed Discharges at Atmospheric Pressure and Higher

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Fourth Annual Review Meeting of the **AFOSR MURI** "***Fundamental Mechanisms, Predictive Modeling, and Novel Aerospace Applications of Plasma Assisted Combustion***"

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Objectives (Thrust 2)

- Development and validation of detailed low-temperature plasma fuel oxidation and ignition mechanisms, starting with simple fuels and proceeding to surrogate fuels
- Development of reduced plasma chemical fuel oxidation, ignition, and flameholding mechanisms which can readily be incorporated into predictive multi-dimensional reacting flow codes
- Identification of specific processes critical to the enhancement of basic combustion phenomena by nonequilibrium plasmas, in particular processes involving radical and/or excited metastable species.

Tasks

- **Reaction kinetics studies** with spatially controlled plasma discharges at atmospheric pressure and higher
- **Effect of nanoparticle/catalyst** coupling with plasma enhanced combustion in flow reactors and flames

Approach – Reaction Kinetics

- ***Flow reactor*** experiments between pressures from 0.1 and 3 bar and a temperature range of ambient to 1200 K (quartz chamber).
- Perform ***dilute hydrocarbon oxidation experiments*** in excess nitrogen, argon, or oxygen to minimize the temperature rise of reaction and stretch the reaction spatially over a significant length.
- ***Perturb the reaction at different extents of reaction*** with a spatially defined plasma discharge.
- Measure ***temperature and product species*** by sample extraction and GC/FTIR analysis.
- Perform ***kinetic modeling of the reaction kinetics*** with sensitivity and Green's function analysis, $\partial Y_i(x)/\partial Y_j(x')$.

Progress

Flow Reactor Experiments:

- Experiments on the oxidation of C1-C7 alkanes with and without plasma assisted reaction performed for T = 300 – 1250 K and P = 1 atm (*to study interactions of plasma and thermal chemistry*).
- Experiments on H2 oxidation with and without plasma assisted reaction for T = 300 – 1250 K and P = 1 atm (*to study interactions of plasma and thermal chemistry*).
- *In-situ* OH LIF measurements in progress with and without plasma for H2 oxidation in collaboration with OSU (Walter Lempert and Igor Adamovich) (*to provide more constraints on model development and link data to other research groups*)
- Plasma assisted kinetics experiments with O2/N2/Ar mixtures as a function of temperature were conducted to quantify NxOy formation in the presence and absence of fuels (*to determine the levels of NOx, rates of oxides of nitrogen formation, and potential interaction with hydrocarbon chemistry in flow reactor experiments with N2*).
- Experiments initiated with nanometer aluminum and carbon particles introduced into flow stream coupled with plasma.

Kinetic Modeling:

- Collaborations were continued with OSU (Igor Adamovich) to model the PSU ethylene oxidation experiments with Hai Wang's mechanism.
- Plasma model coupled to SENKIN and CHEMKIN with formal sensitivity analysis.

Plasma Flow Reactor

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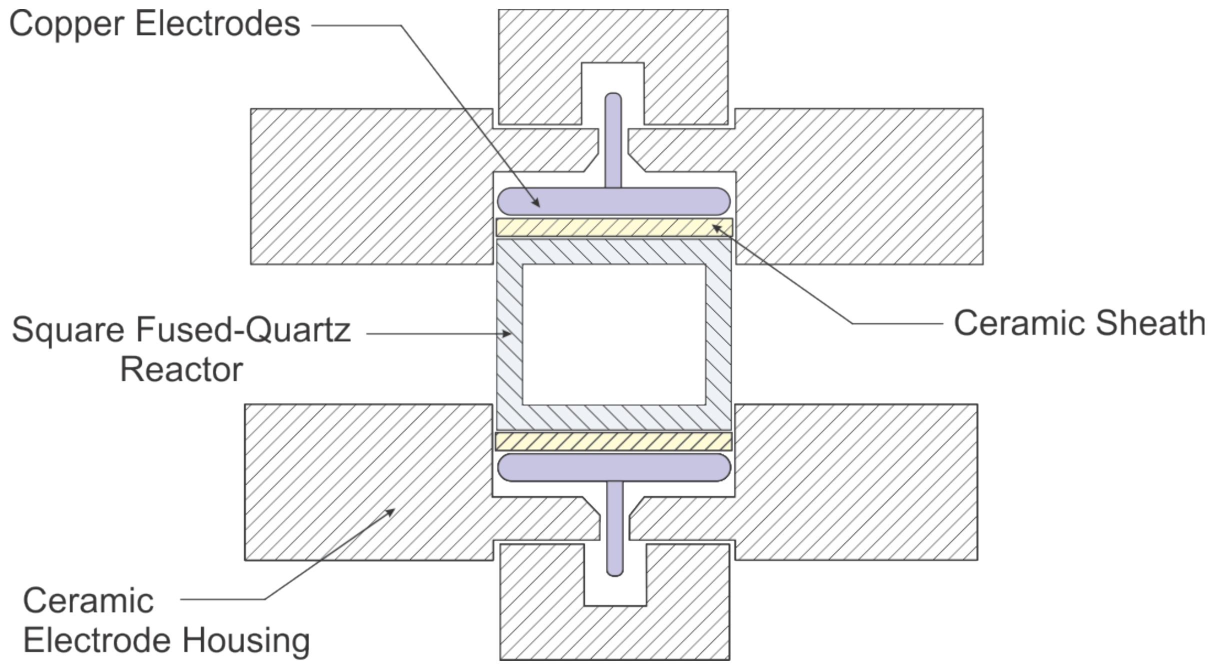
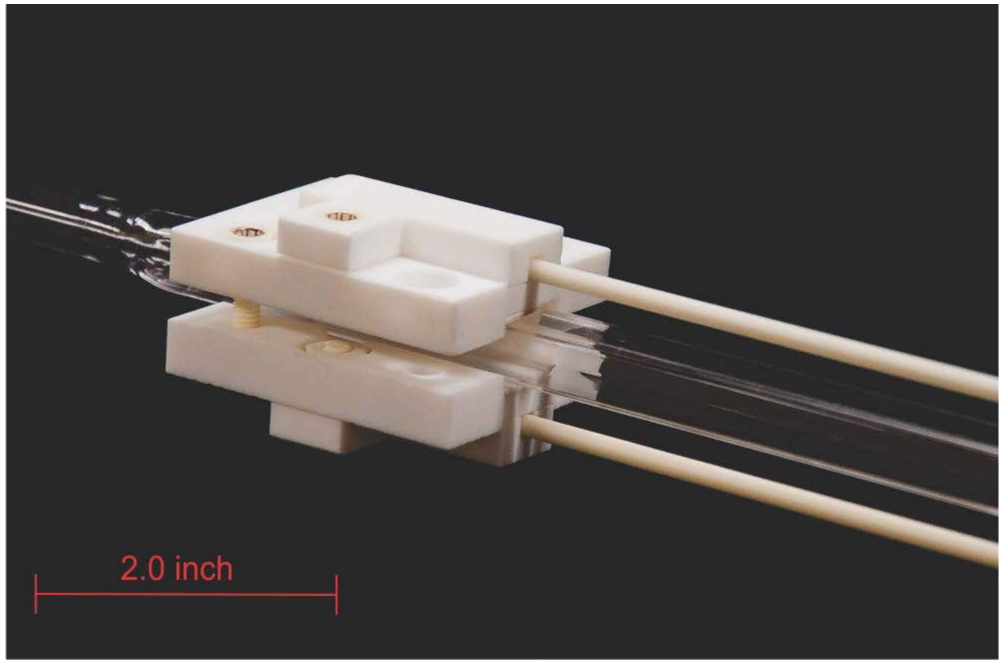
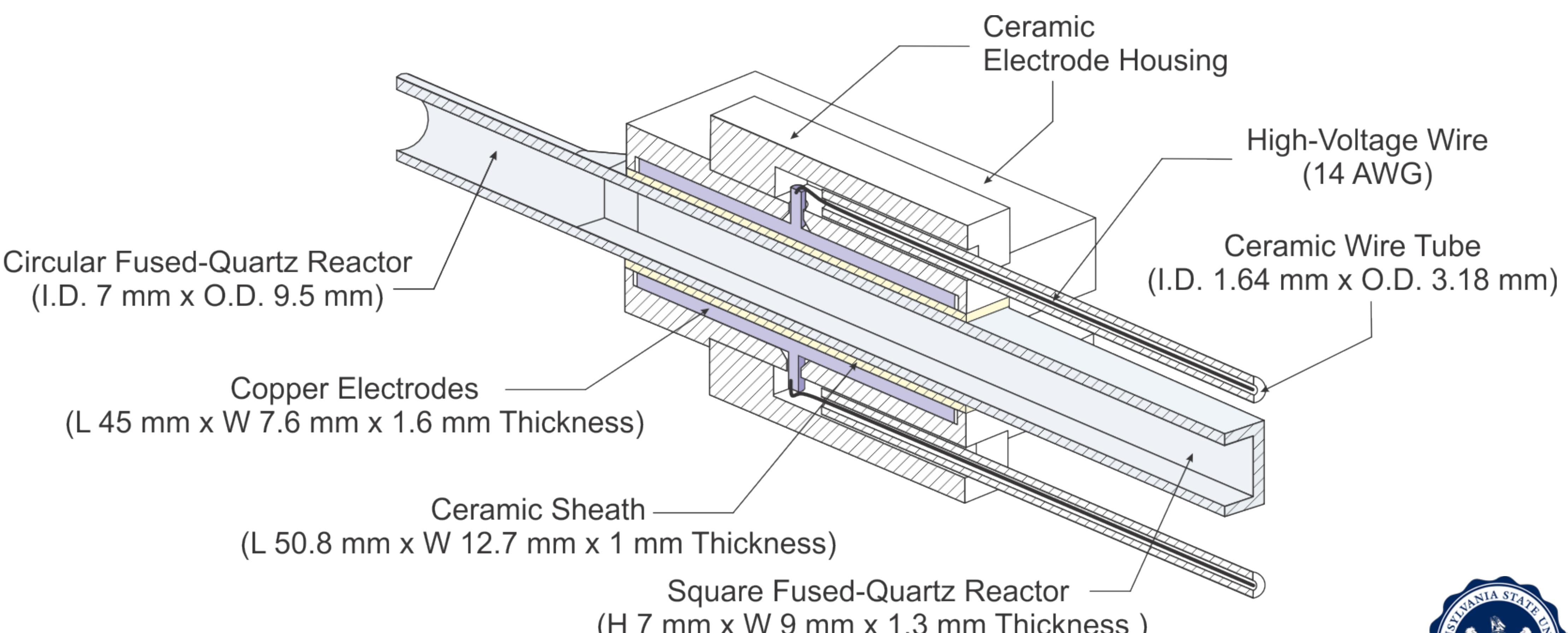
Plasma Discharge Assembly



Cross-Section View (Front)



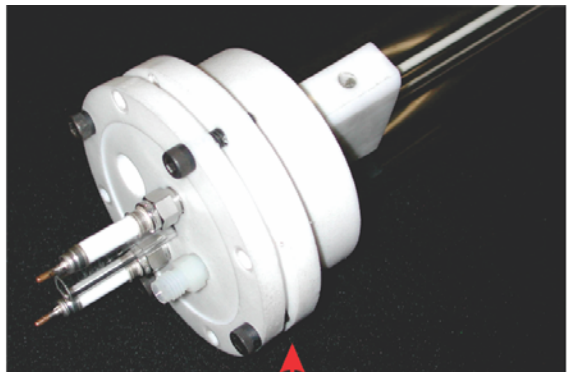
Actual Image of Assembly



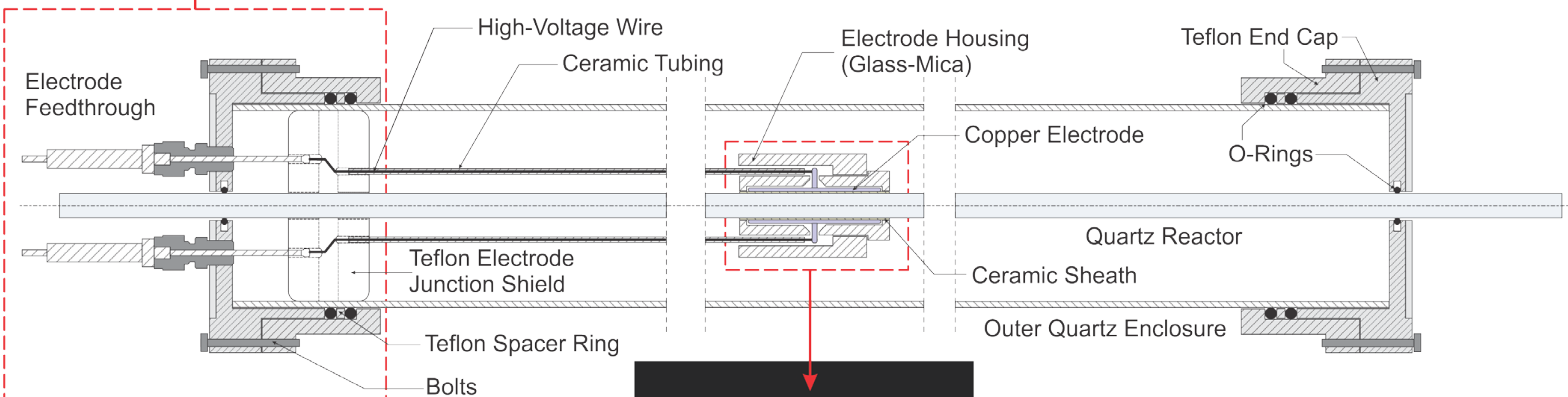
Cross-Section View (Isometric)

Plasma Flow Reactor Assembly

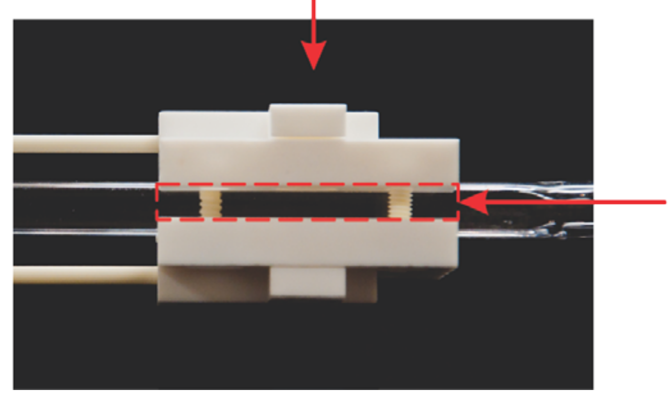
Actual Image Teflon End Caps w/ Electrode Feedthroughs



- The **plasma discharge assembly** is housed in a quartz tube (58mm I.D.) with Teflon end caps
- Actual flow reactor length is 1524 mm (60 in.)



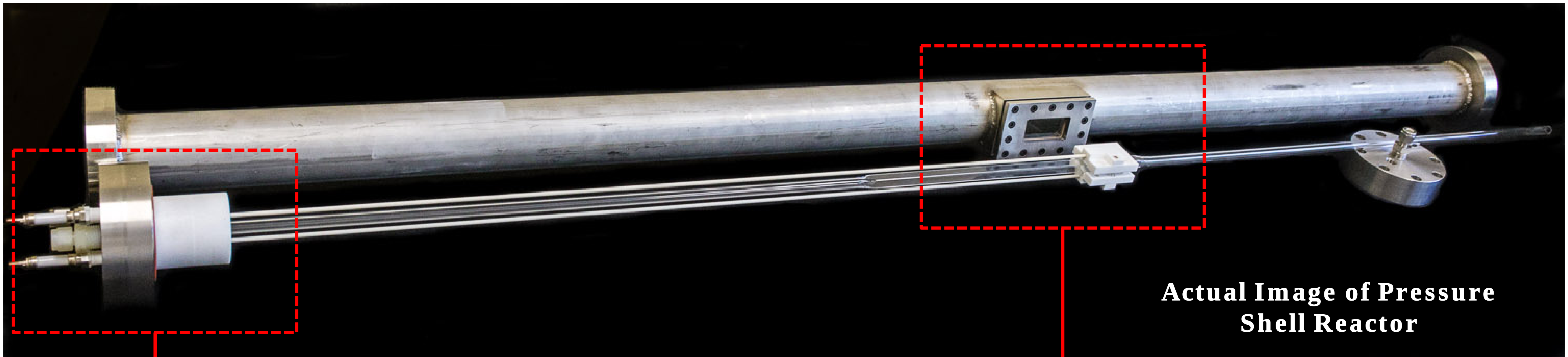
- The **copper electrodes** are connected to ceramic insulated terminal feed-throughs



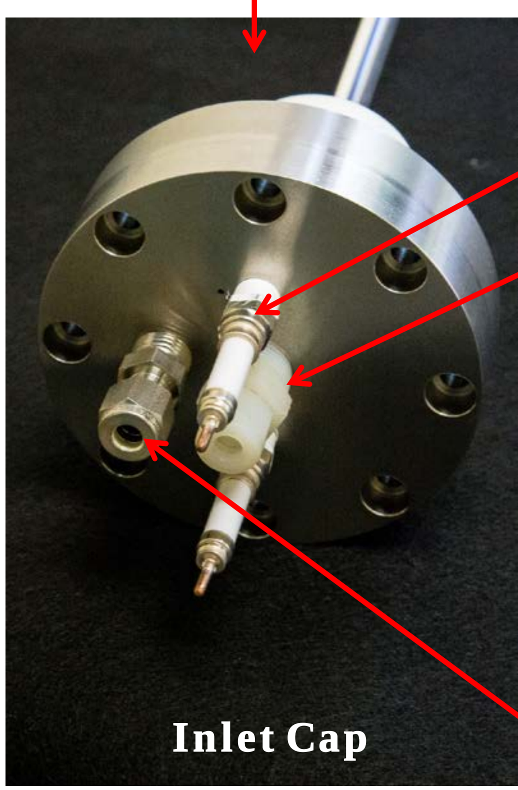
Actual Image Discharge Assembly (Side View)

Reactor Assembly for 1 < P < 3 atm

- Modular plasma discharge reactor can be interchanged with redesigned pressure shell to perform kinetic studies at P > 1 atm



Actual Image of Pressure Shell Reactor

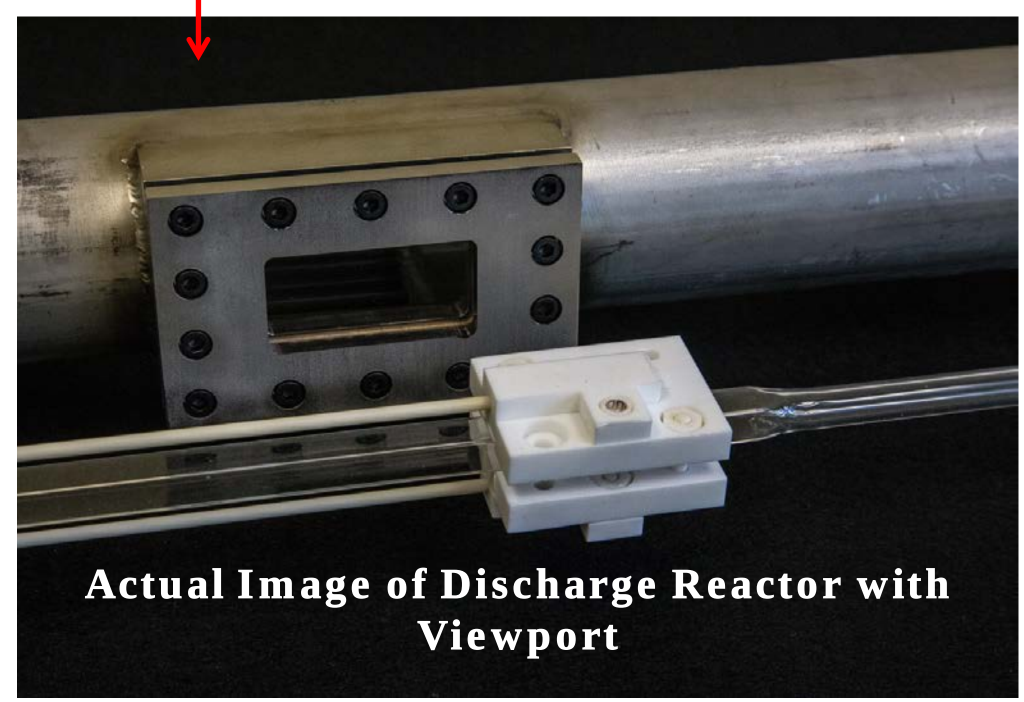


Inlet Cap

Electrode Feedthroughs

Gas Mixture Inlet

Pressure Purge Gas Inlet

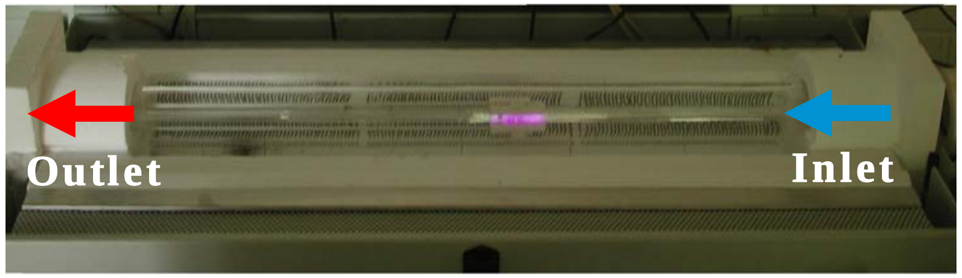


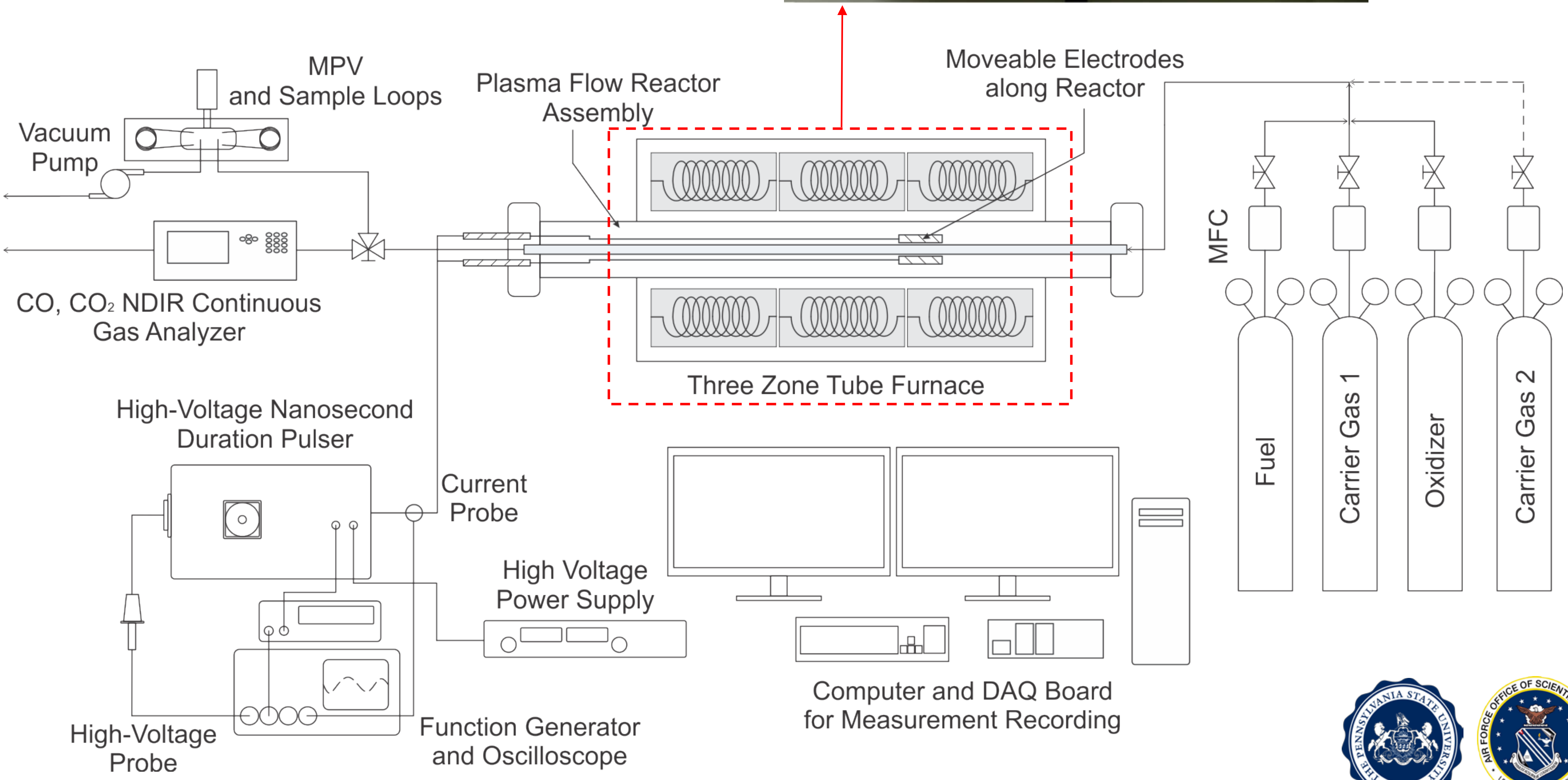
Actual Image of Discharge Reactor with Viewport

Plasma Flow Reactor Experimental Facility

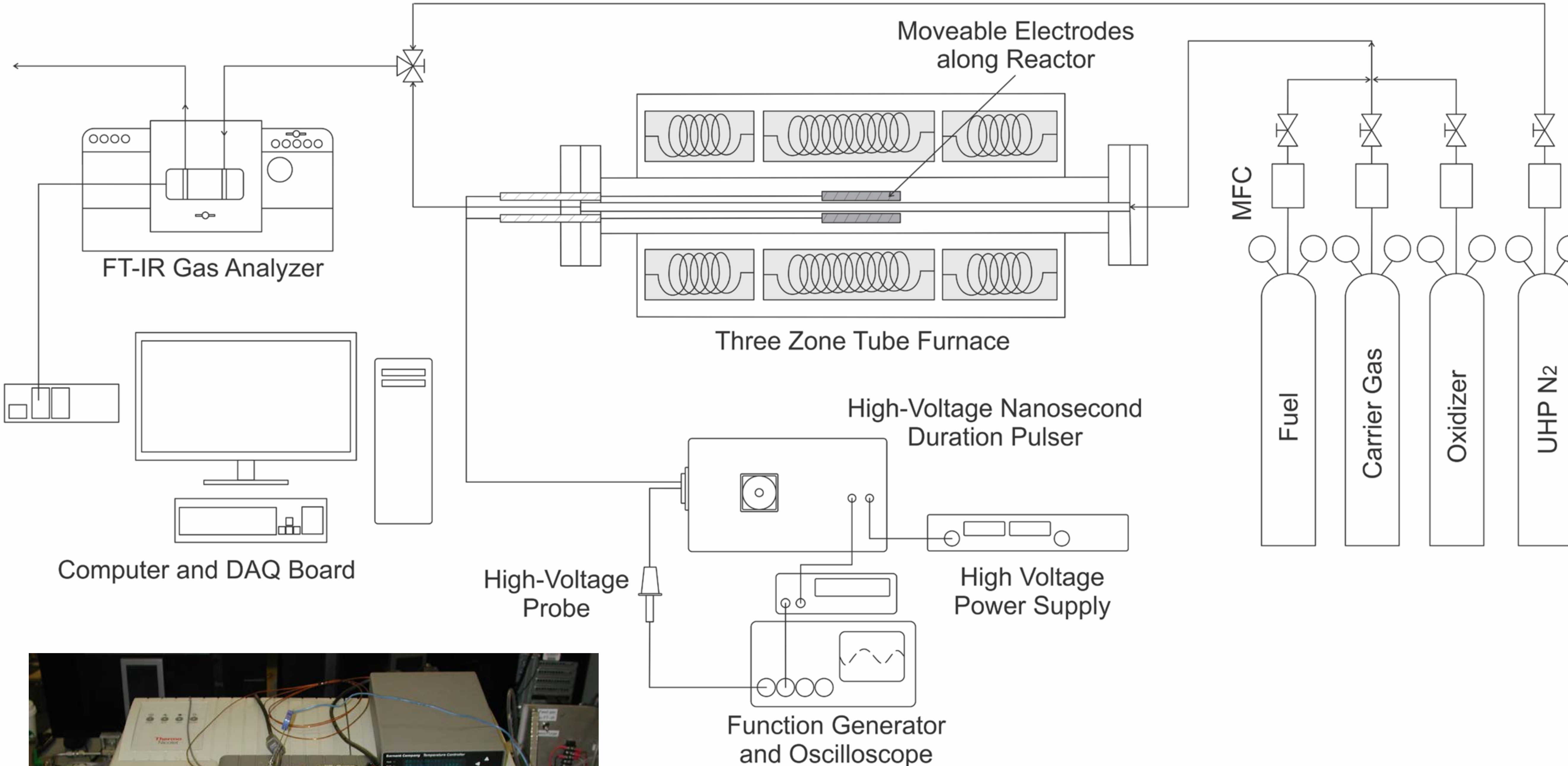
- Heating is achieved using a **three-zone tube furnace** with a length 609 mm (24 in.)
- Product species analysis performed using **NDIR** (online) and **Gas Chromatography** (offline)

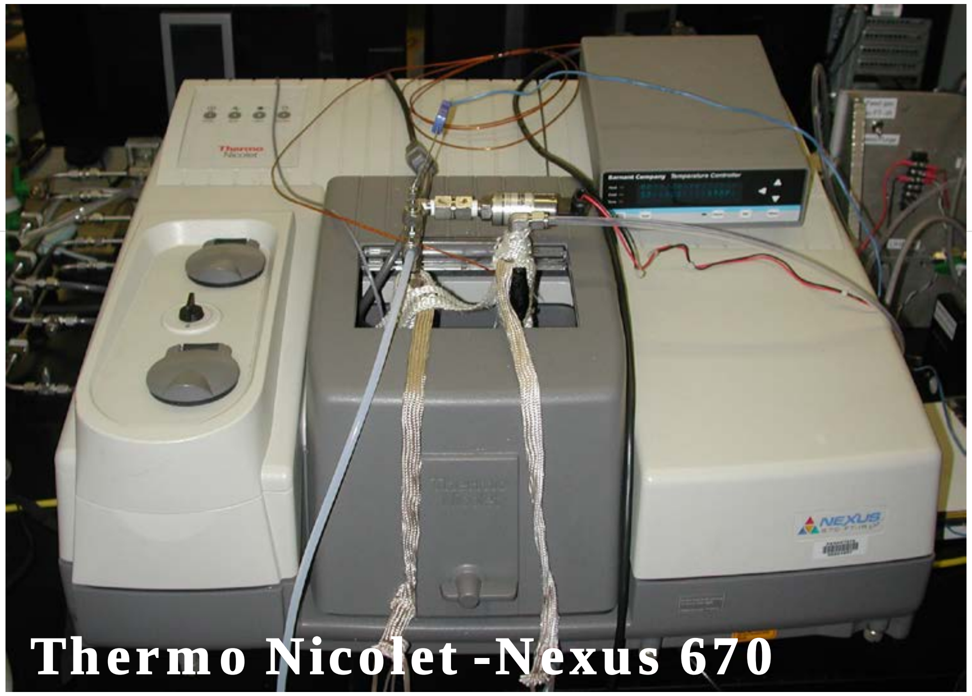
Actual Image Tube Furnace w/ Plasma Flow Reactor





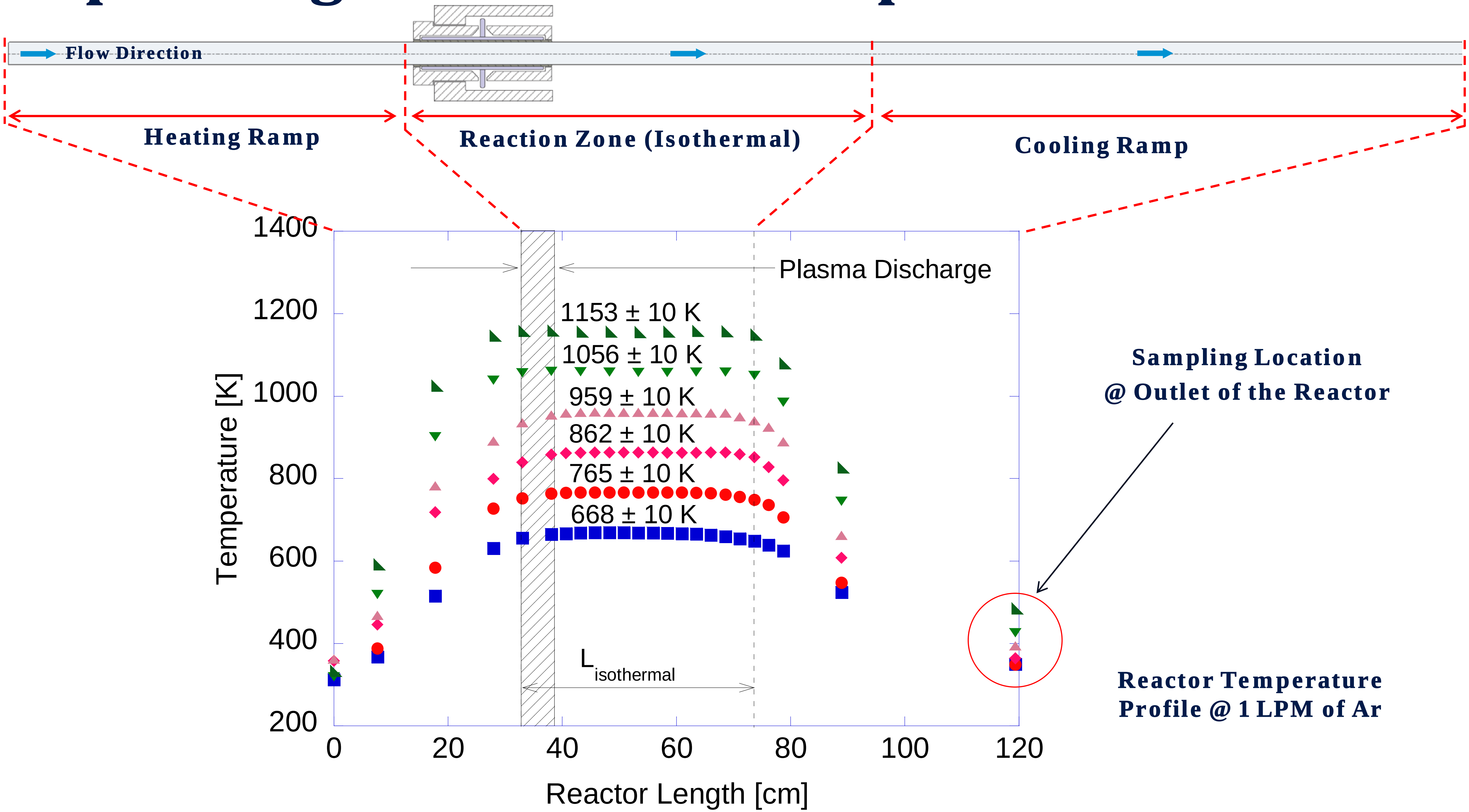
Experimental Facility – cont'd





- **FTIR** (online) connected to exhaust stream of reactor
- Capable of detecting hydrocarbon and nitrogen-containing species (i.e. NO, NO2, N2O)

Operating Conditions - Temperature



- The measured temperature profiles are used to establish an empirical correlation with the tube furnace temperature over a range of 350 K < T_{iso} < 1250 K. The correlation is used for kinetic modeling
- Experiments verify that heating from the plasma discharge is negligible in the diluent flow
- In Ar/O2 flow, ~ 10 K increase in gas temperature observed at the maximum voltage setting for a given reactor temperature of 814K

Operating Conditions - Plasma Discharge

Experimental Conditions: P = 1 atm, Q = 1 LPM Ar, Gate = 10 μsec

Plasma Discharge : Vplasma = 15 kV, ν = 1 kHz

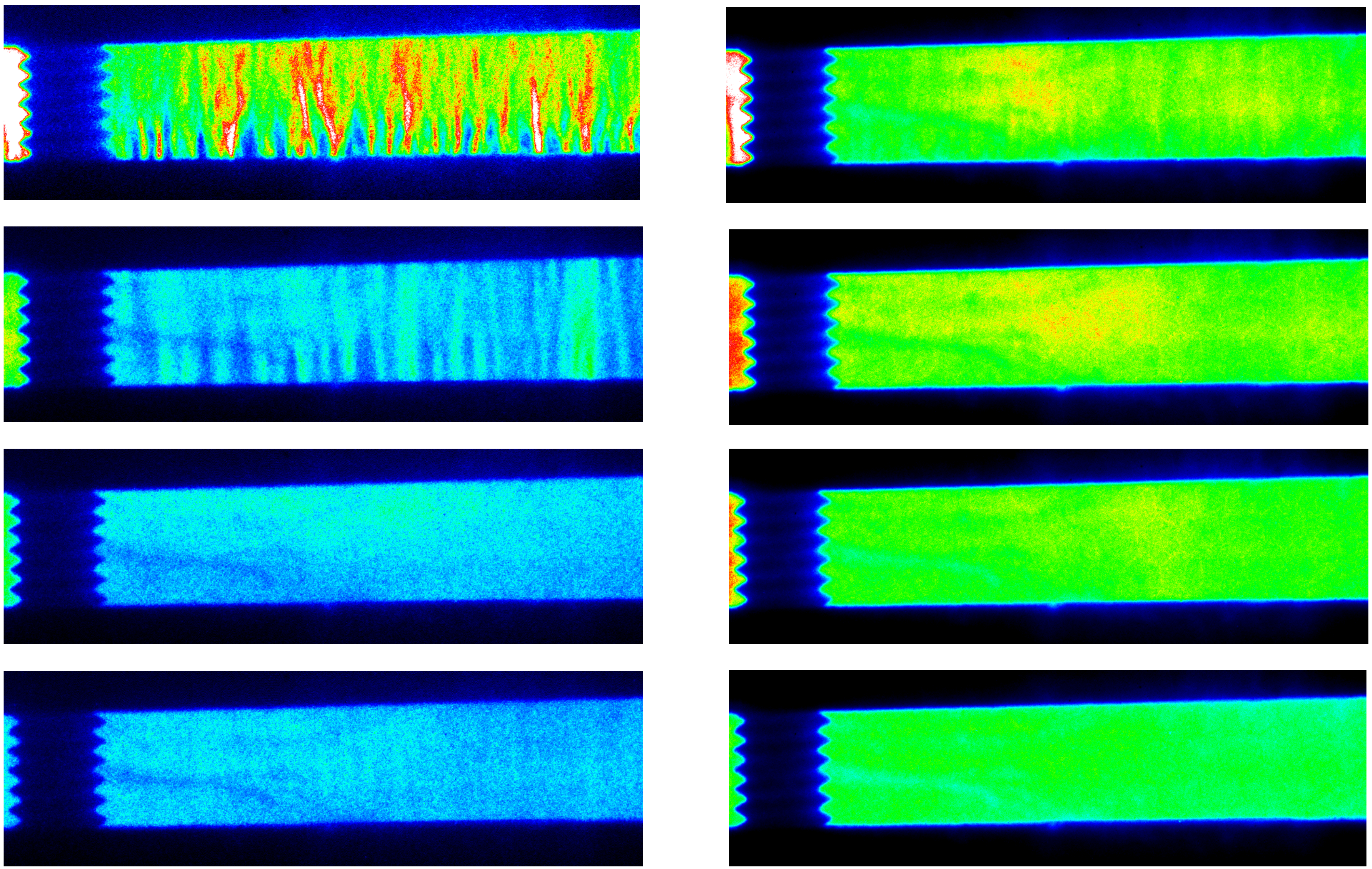
ICCD Single Pulse Images | **ICCD Multiple Images (average of 50)**

298 K

427 K

620 K

814 K

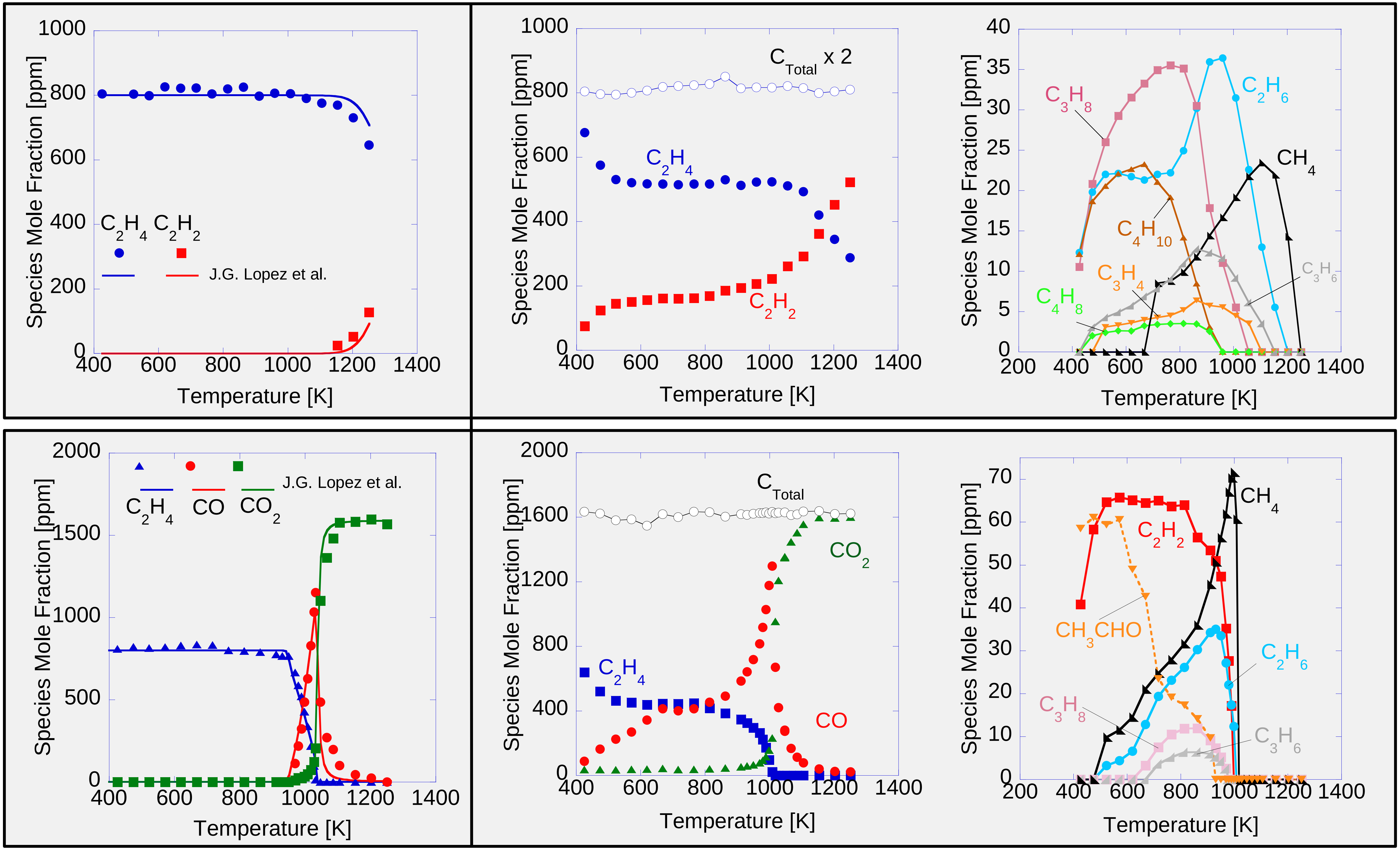


Brief Review of Previous Results

C_2H_4 Pyrolysis and Oxidation Experiments

Thermal | **Plasma Assisted**

Pyrolysis / **Oxidation**



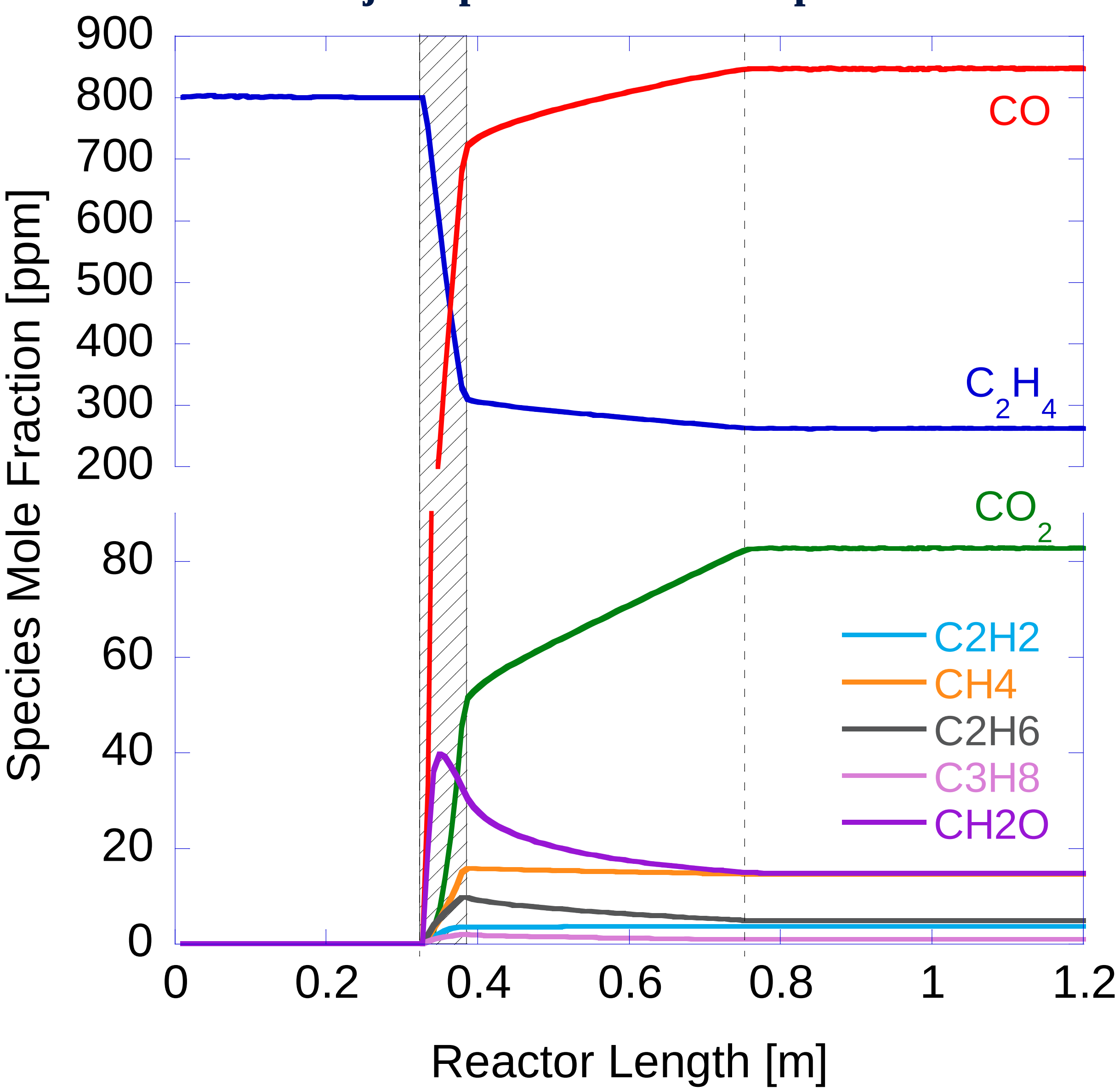
Experimental Conditions: P = 1 atm, Q = 1 LPM, 800ppm C2H4/3000ppm O2 / Ar by balance

Plasma Conditions: 10 kV, v = 1 kHz

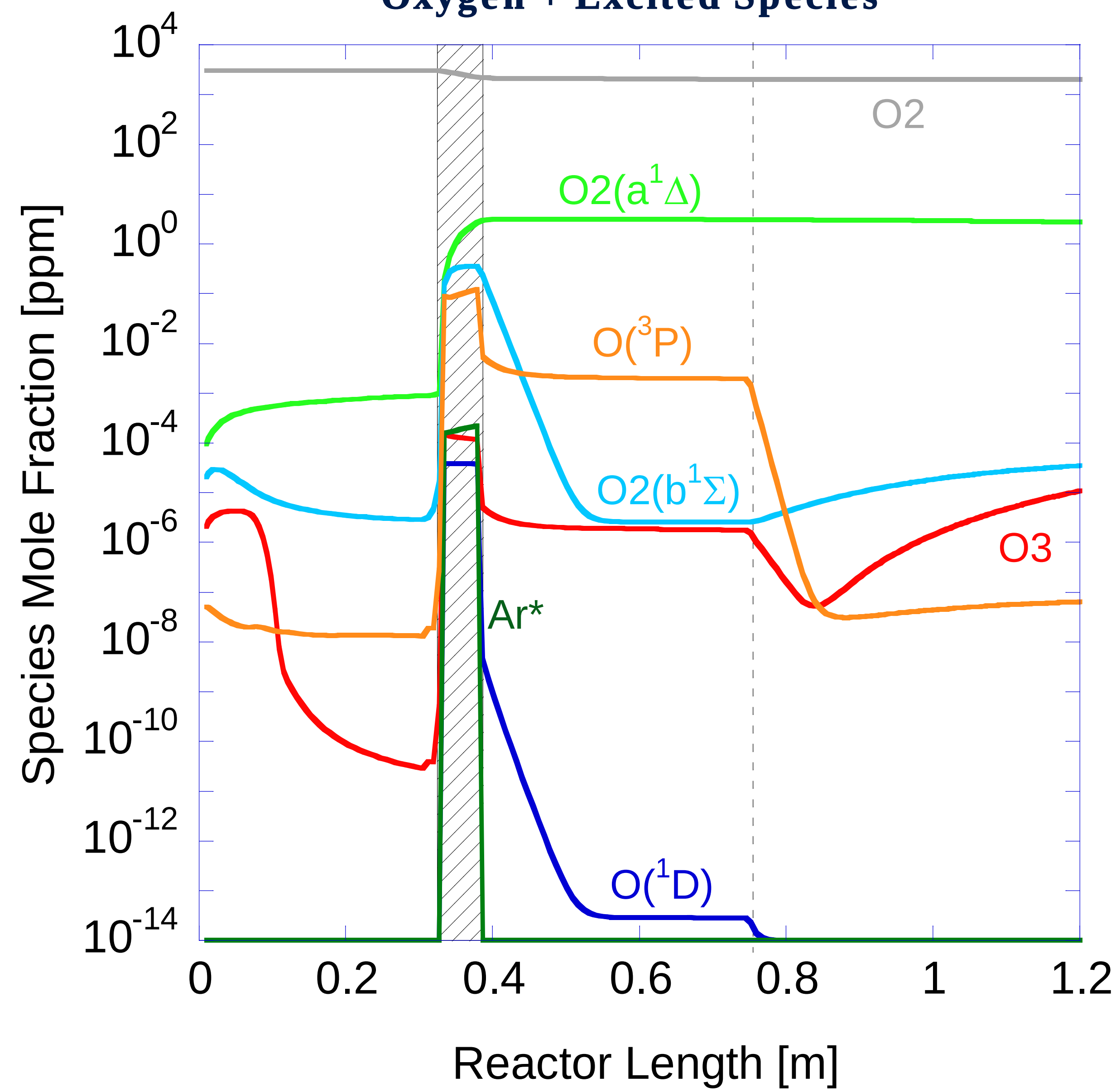
Modeling Results for a Single Experiment

Modeling Conditions: P = 1 atm, Q = 1 LPM, T = **863 K**, 800ppm C2H4 / 3000ppm O2 / Balance Ar
Plasma Conditions: 0.043 meV/molecule/pulse

Major Species + Minor Species



Oxygen + Excited Species



- Most of the reaction is isolated in the plasma region, with some further reaction occurring in the isothermal region
- Experimental results correspond to species concentration at the exit of the reactor

C_1-C_7 Alkanes Plasma Assisted Oxidation

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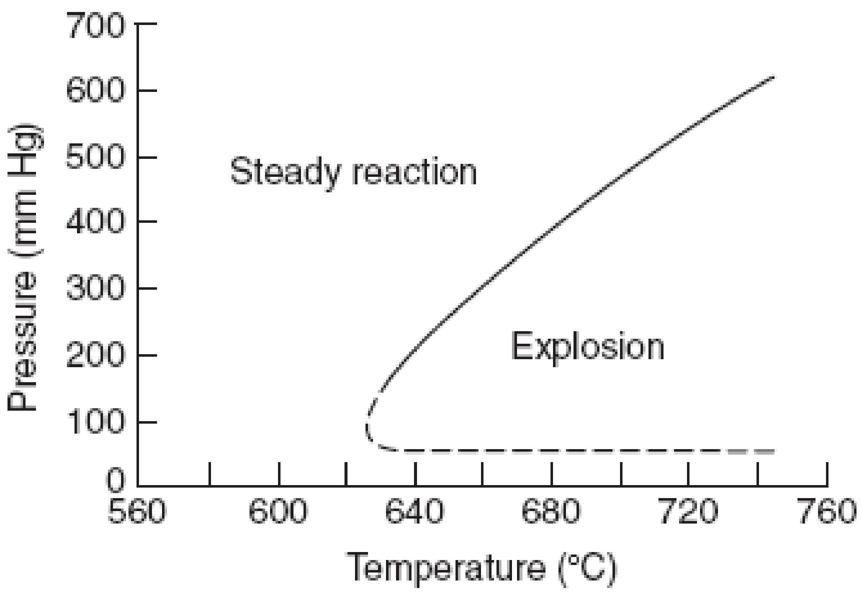
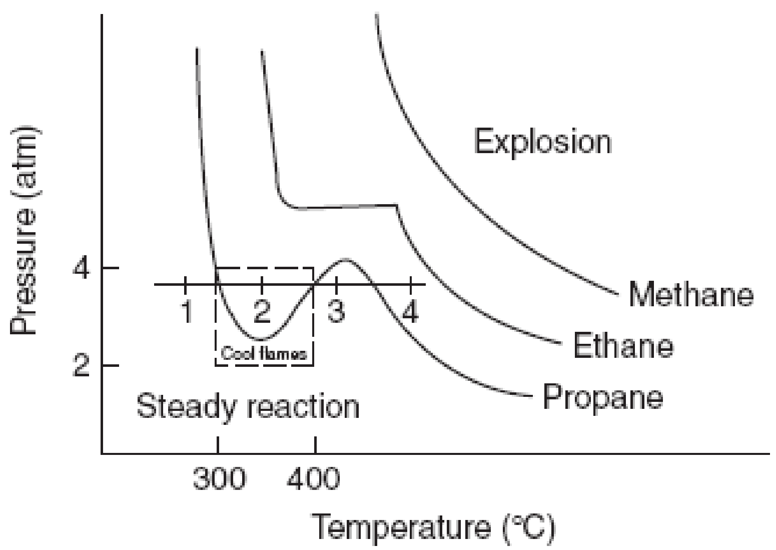
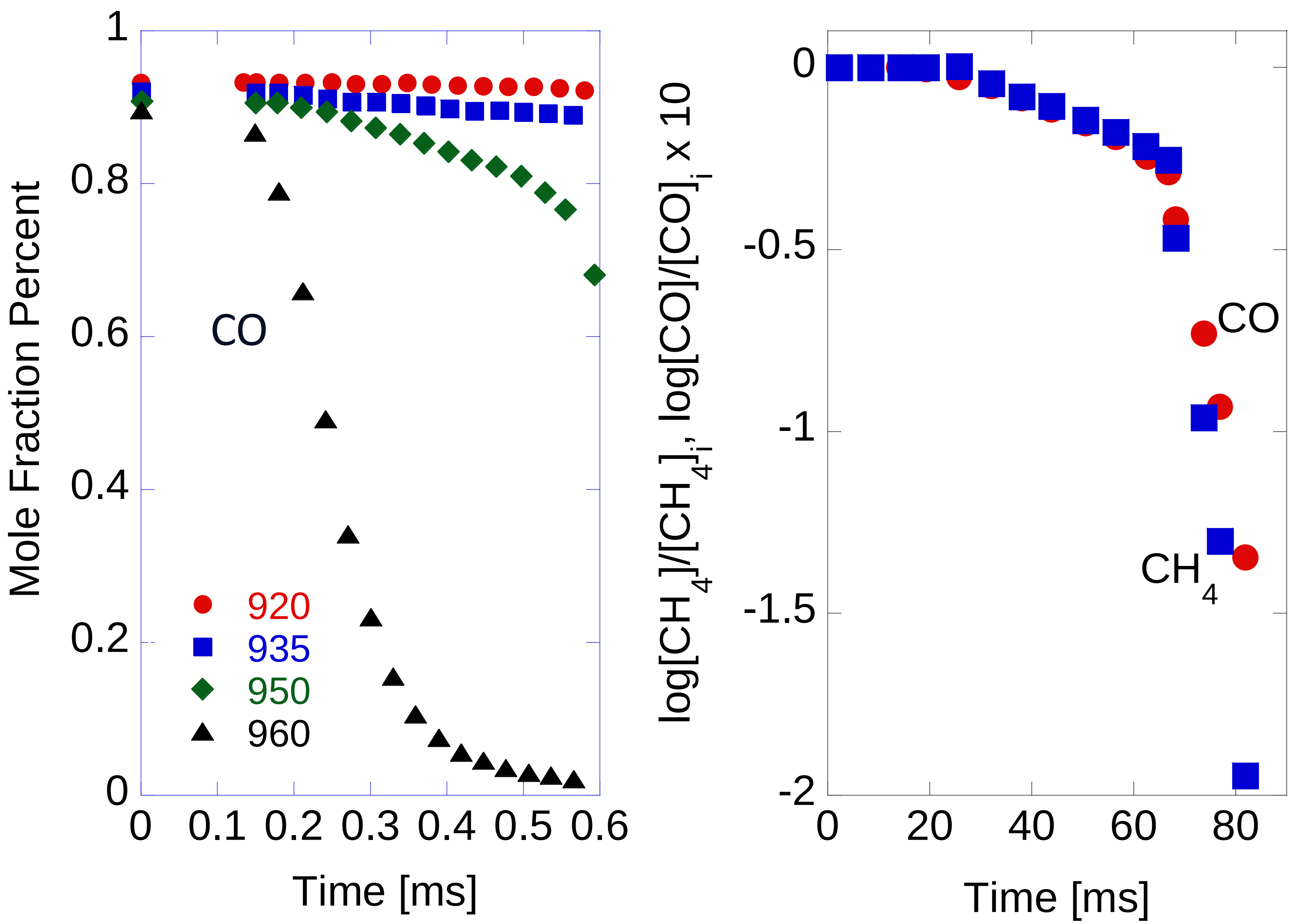
Thermal Explosion Limits





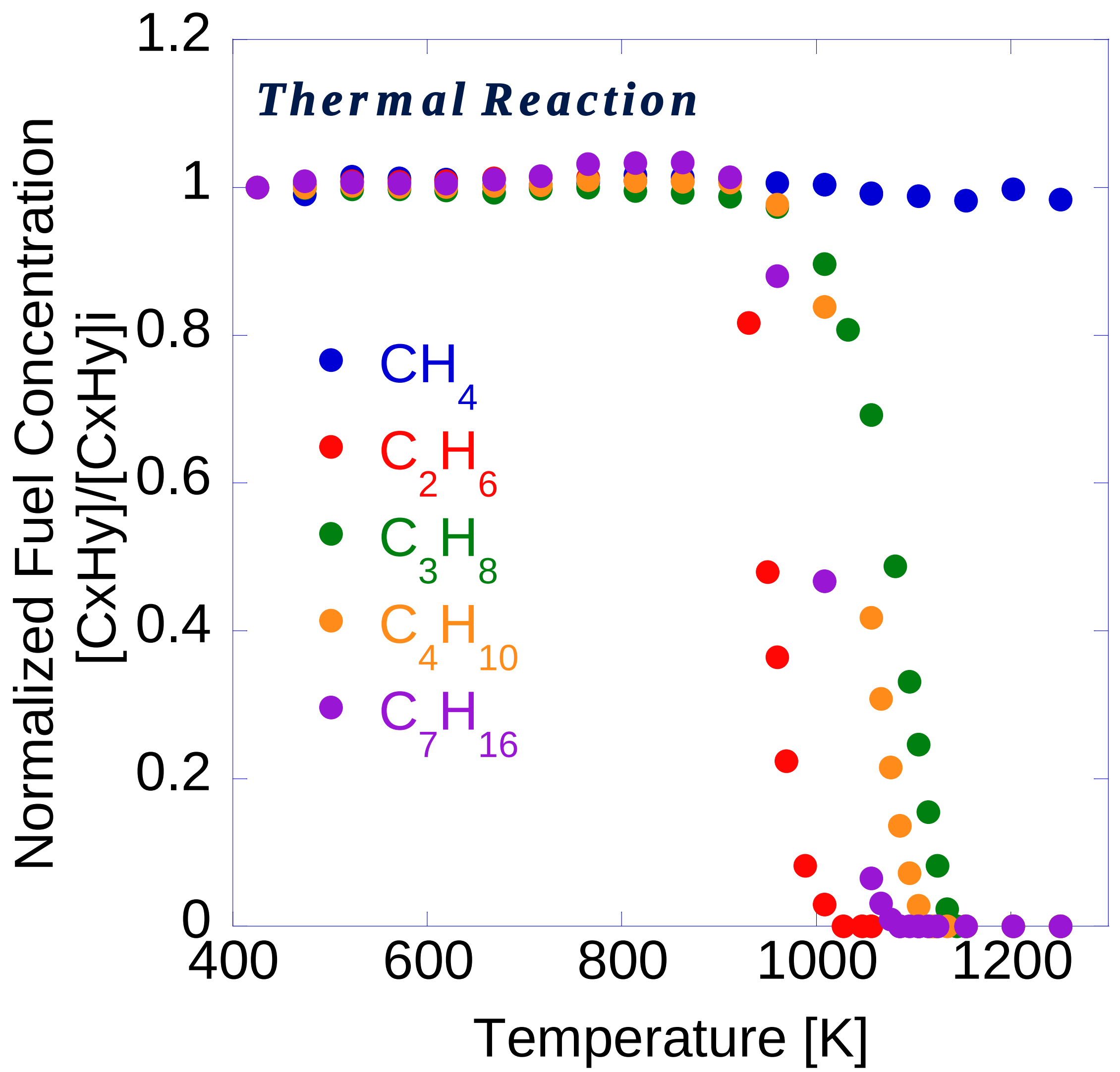


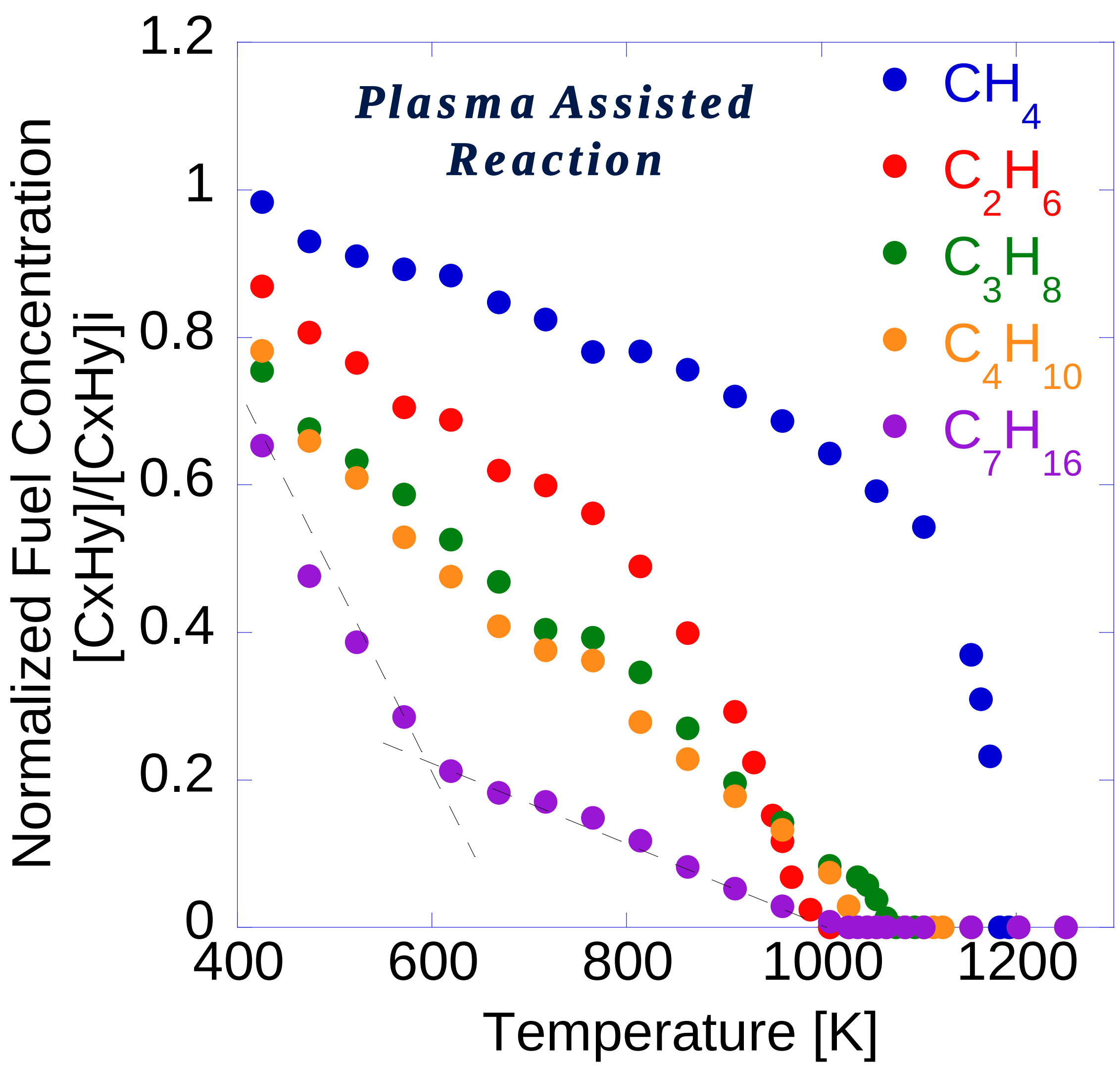
I. Glassman and R.A. Yetter, **Combustion**, 4th Ed, Academic Press, 2008

Yetter, R.A. and Dryer, F.L., **Proceedings of the Combustion Institute**, 24, 757, 1992.

C_1-C_7 Alkane Fuel Consumption

Experimental Conditions: P = 1 atm, Q = 1 LPM, 1600ppm C*x*H*y*/3000ppm O2 / Ar balance
Plasma Conditions: 10 kV, *ν* = 1 kHz





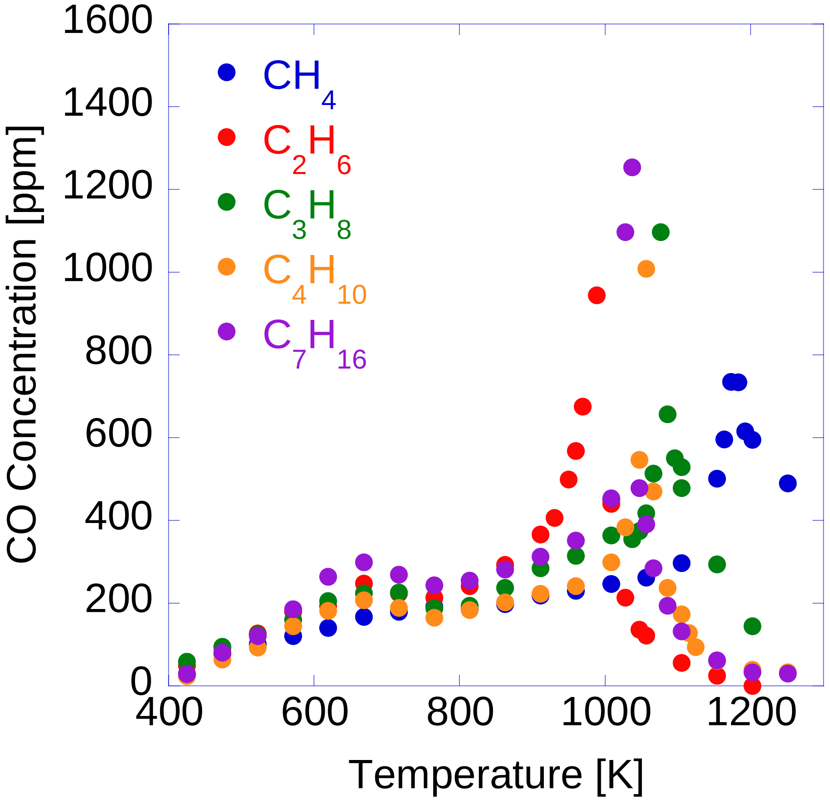
- Without the plasma, onset of fuel consumption occurs for T > 900 K. With the plasma, onset of fuel consumption occurs at T = 420 K for all the fuels.
- The plasma also lowers the temperature for complete consumption of all fuels (the temperature for onset of high temperature ignition).
- For C3 and larger fuels, a transition in reaction rate is observed 600 and 700 K.

Major Intermediate Species Generation

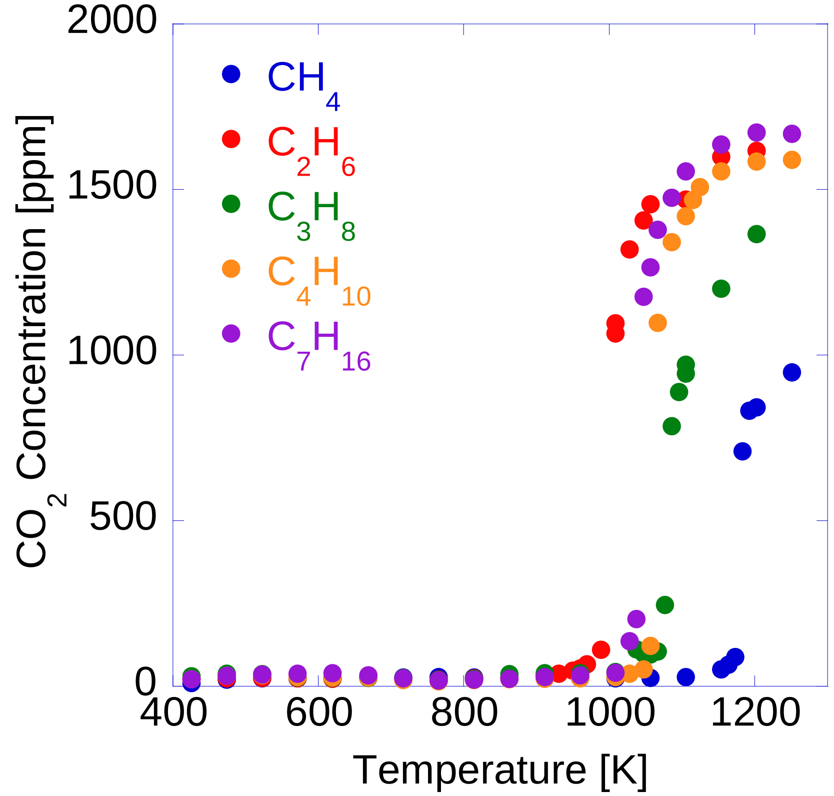
Experimental Conditions: P = 1 atm, Q = 1 LPM, 1600ppm C*x*H*y*/3000ppm O2 / Ar balance

Plasma Conditions: 10 kV, *ν* = 1 kHz

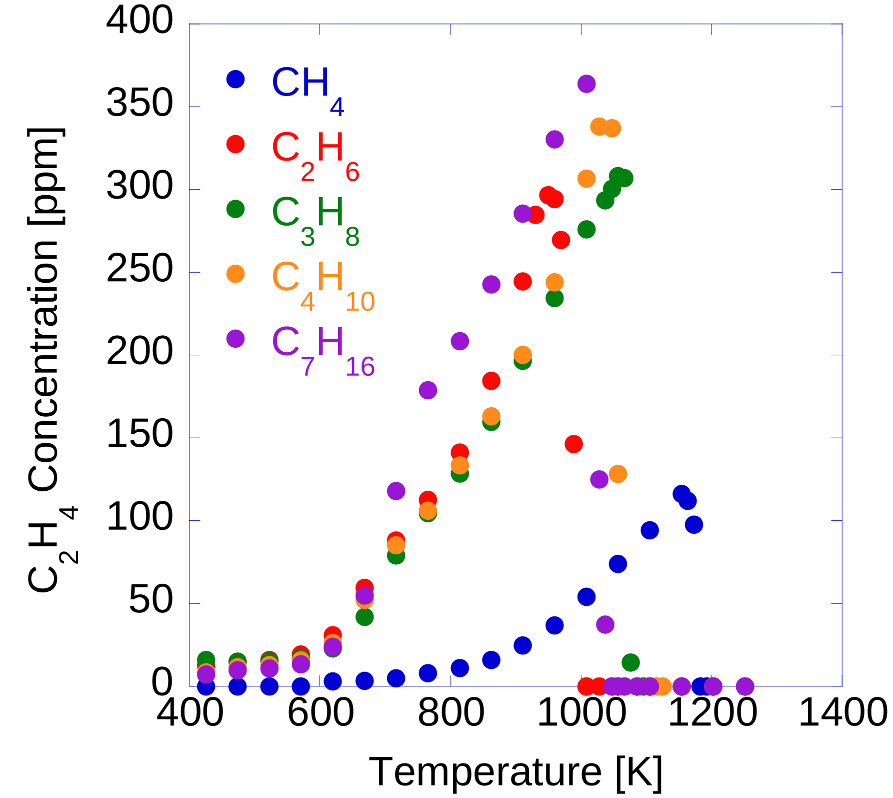
CO



CO2

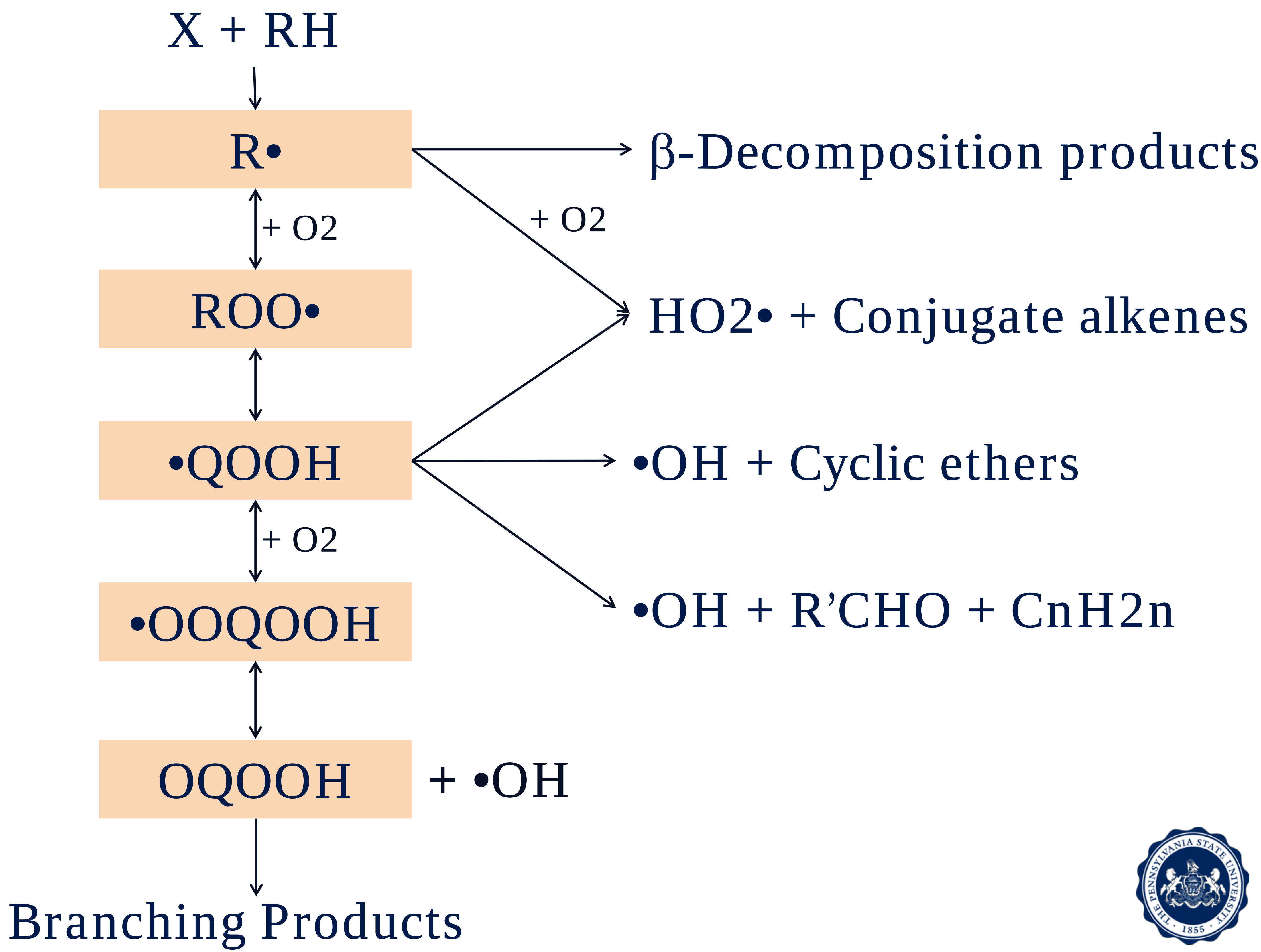


C2H4



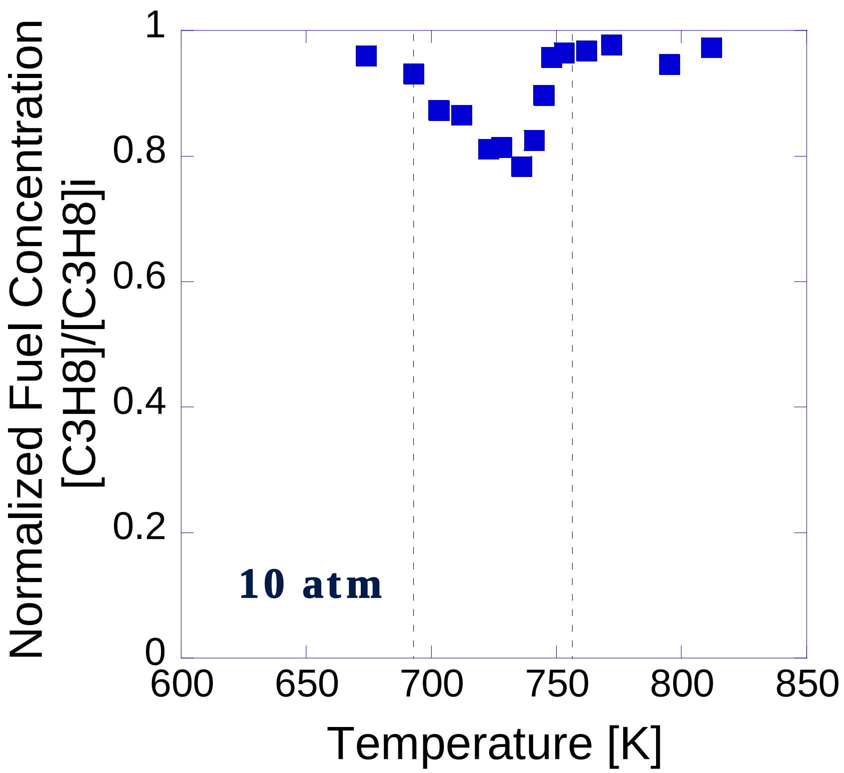
- Before the onset of thermal effects, the plasma has a consistent effect on the formation CO; once the thermal effect is initiated both the plasma and thermal reactions contribute to forming and consuming CO
- Below 960K, very little heat producing CO2 is formed.
- With the exception of CH4, all fuels appear to form similar amounts of C2H4 from 420K until the temperature where C2H4 consumption exceeds its formation.

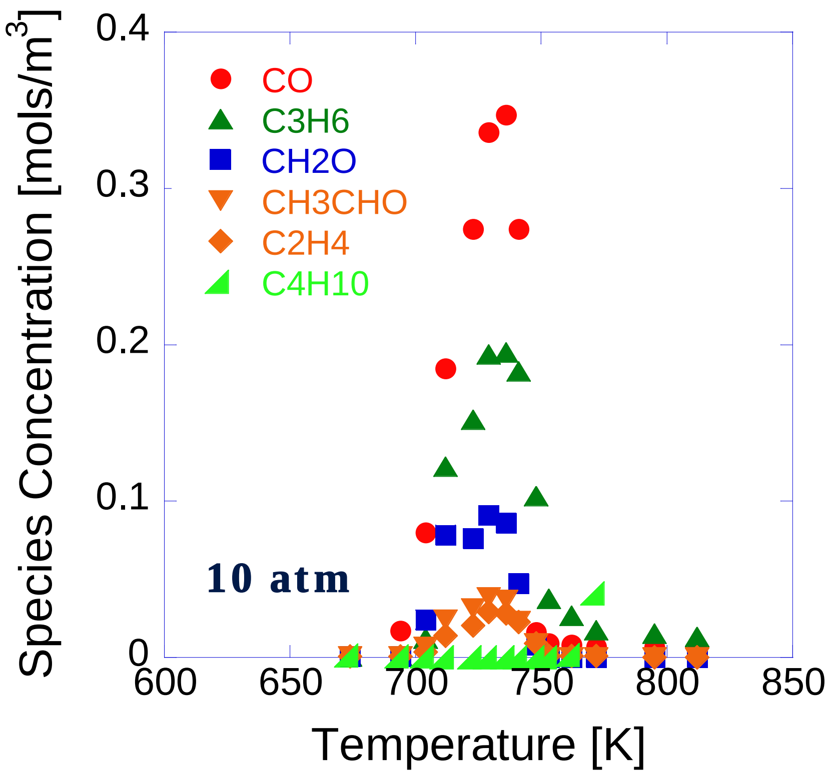
Kinetic Scheme of Primary Oxidation Reactions of n-Alkanes

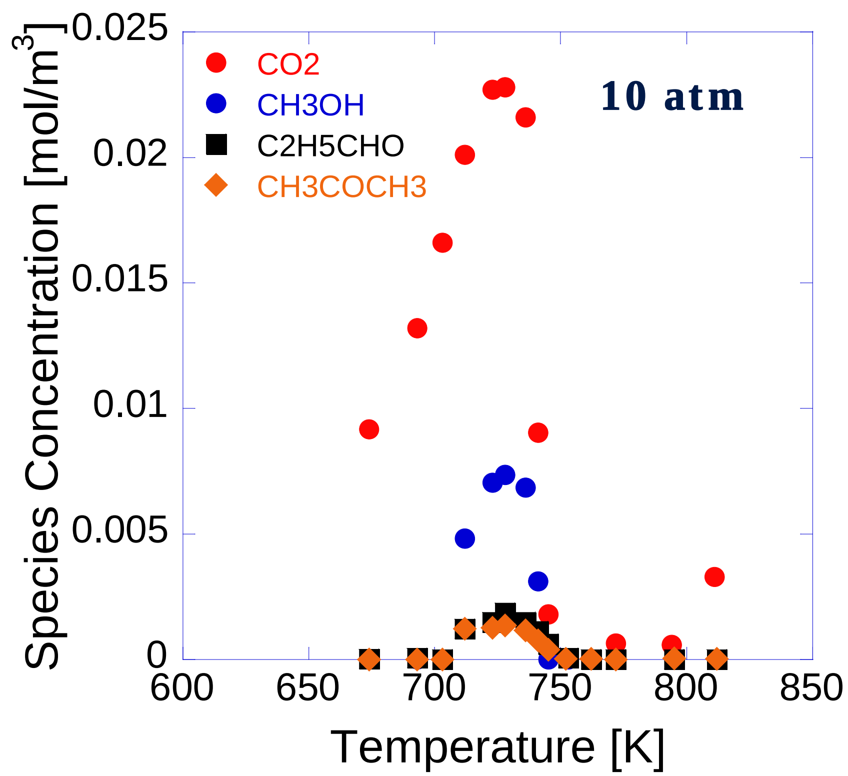


Thermal Oxidation of C_3H_8

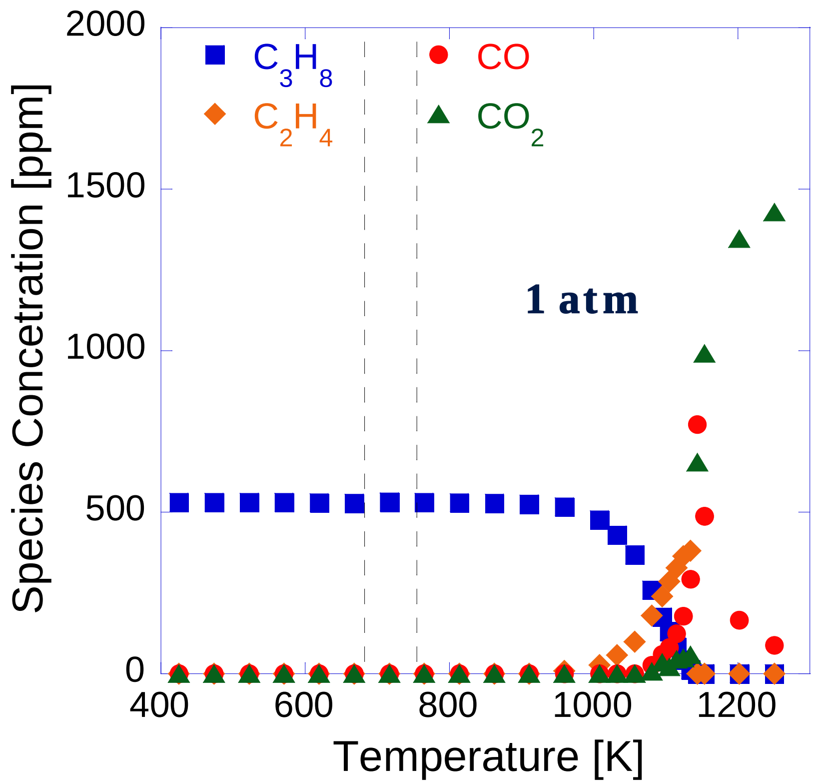
D. N. Koert, D. L. Miller, and N. P. Cernansky, "*Experimental Studies of Propane Oxidation through the Negative Temperature Coefficient Region at 10 and 15 Atmospheres,*" ***Combustion and Flame***, 96, 34-49, 1994.

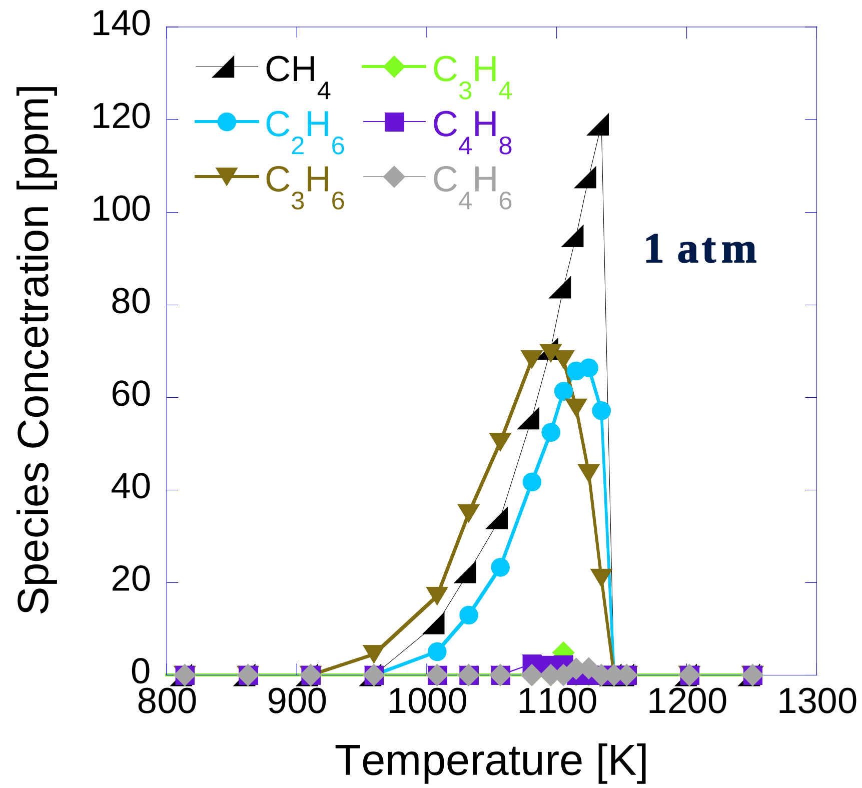






Experimental Conditions: P = 1 atm, Q = 1 LPM, 533ppm C3H8/3000ppm O2 / Ar balance

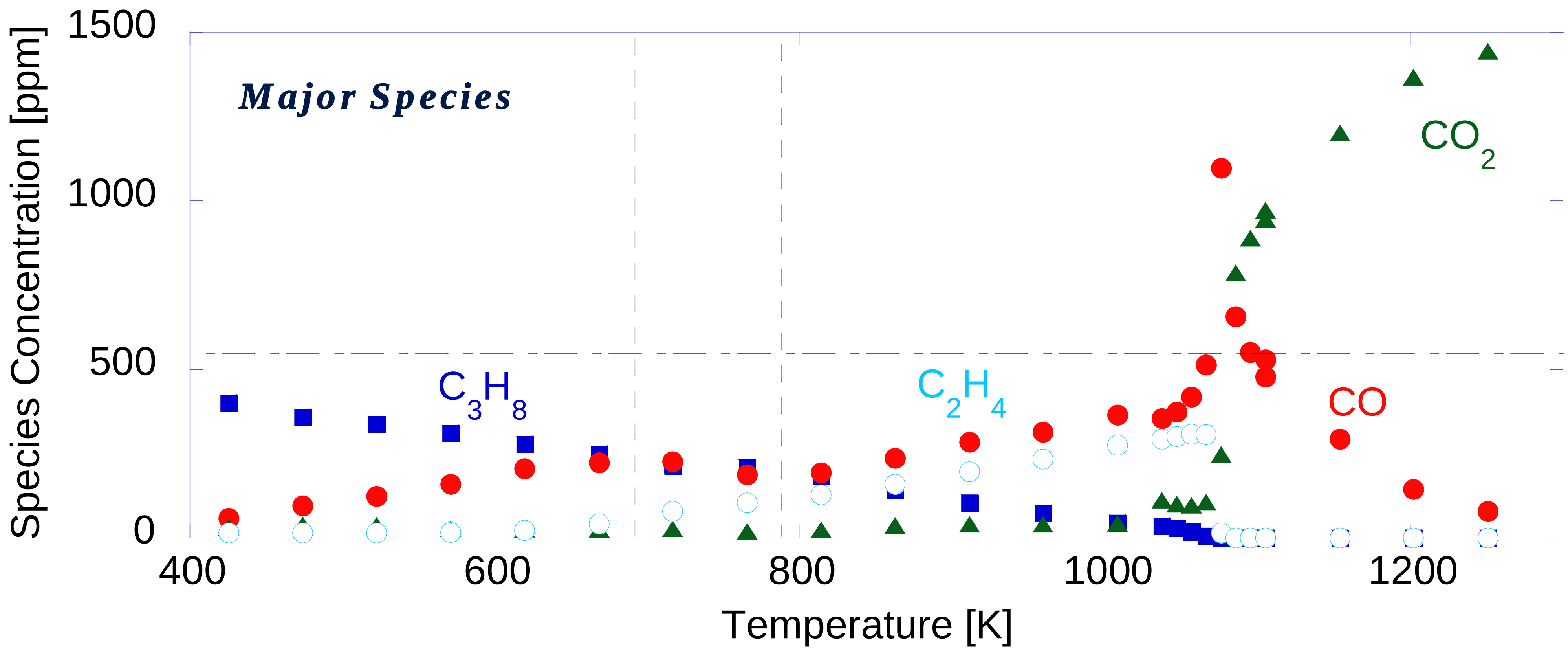


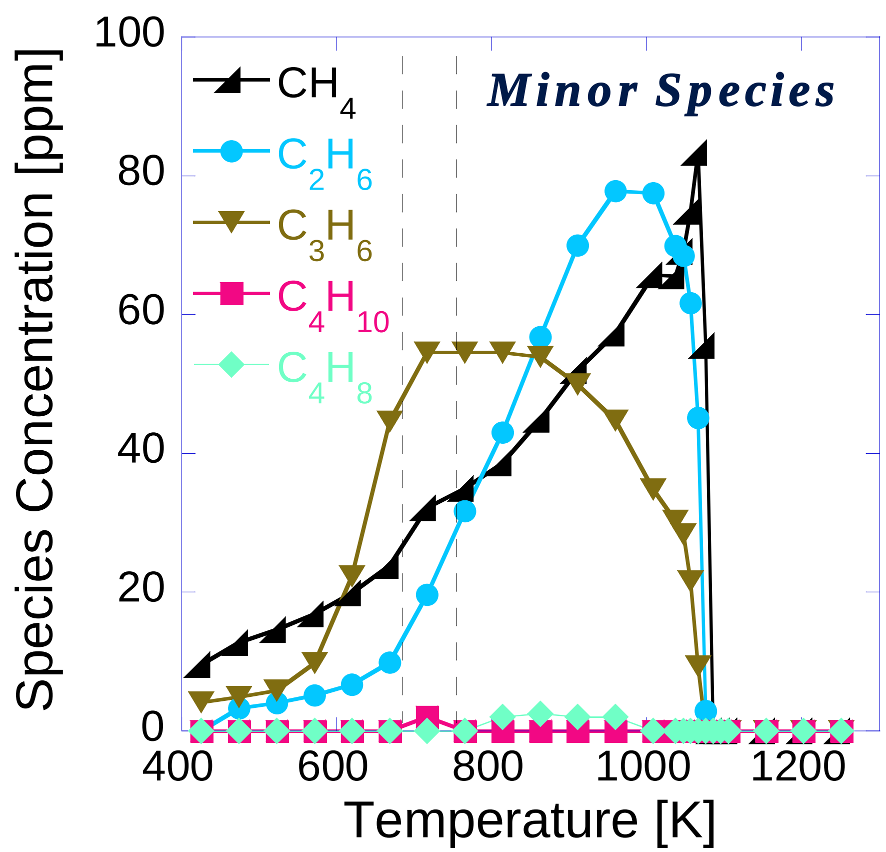


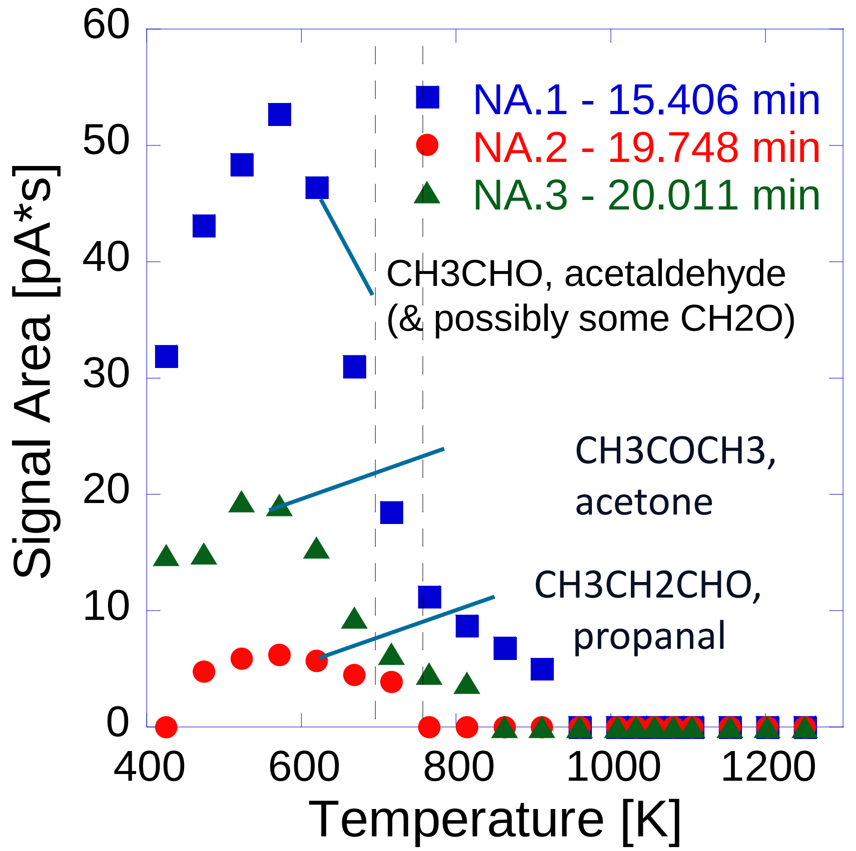
C_3H_8 Plasma Oxidation

Experimental Conditions: P = 1 atm, Q = 1 LPM, 533ppm C3H8/3000ppm O2 / Ar balance

Plasma Conditions: 10 kV, ν = 1 kHz

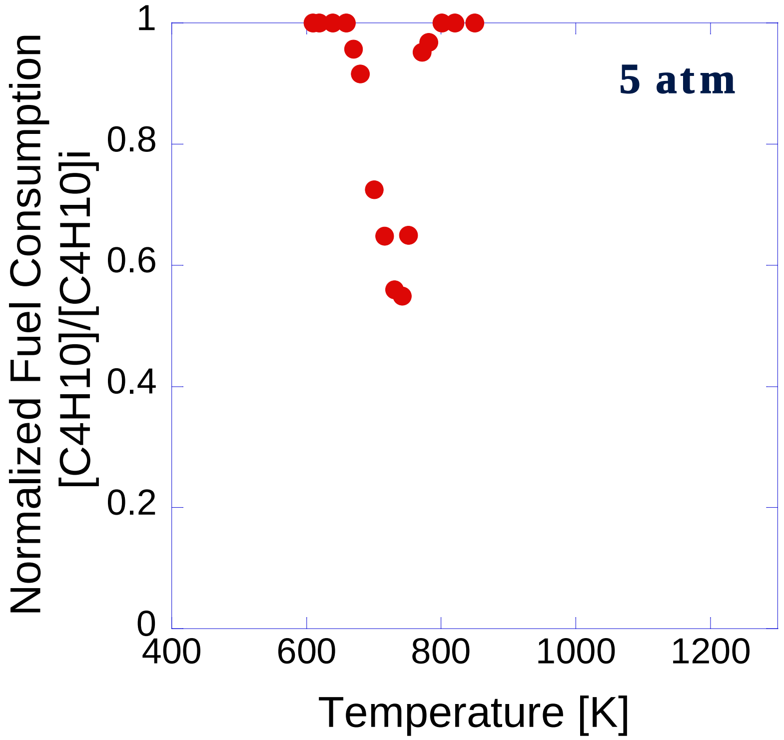




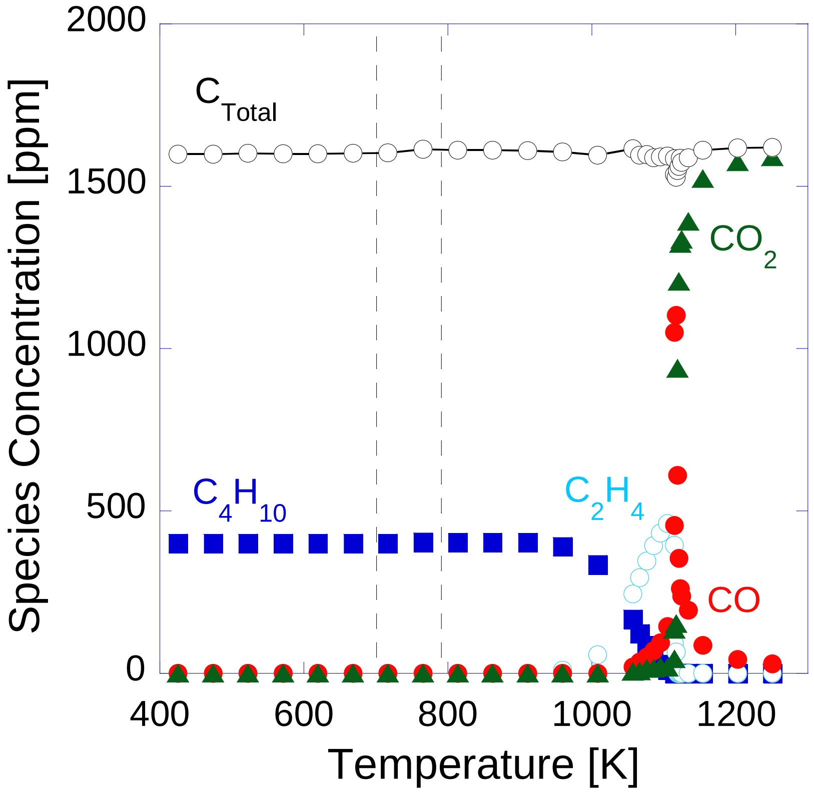


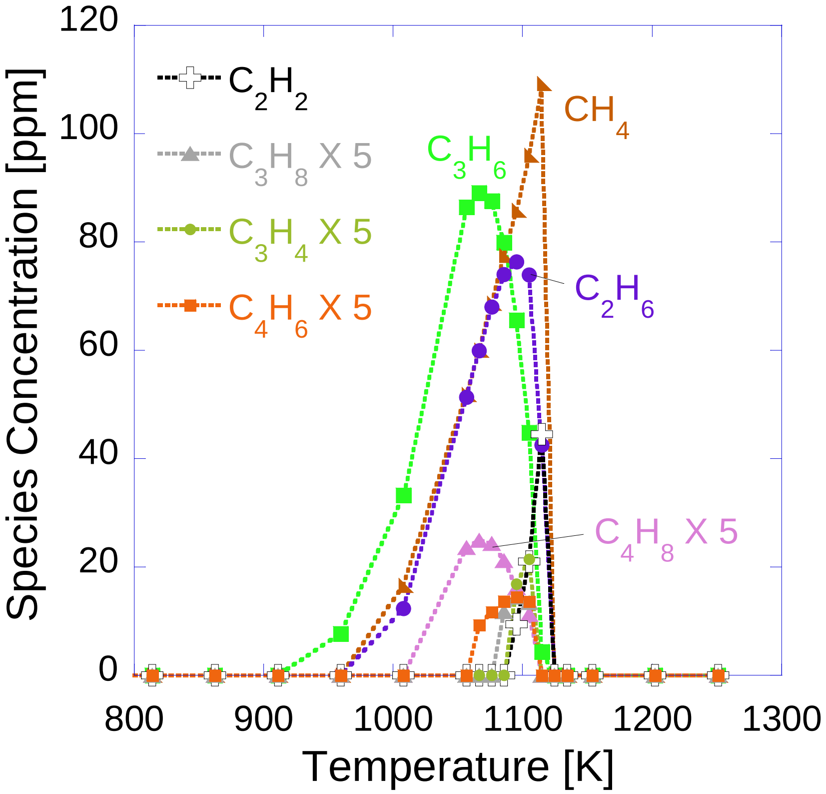
Thermal Oxidation of C_4H_{10}

Anand, A., "*An Experimental Study of n-butane Oxidation at Elevated Pressures in the Low and Intermediate Temperature Regimes*," **MS Thesis**, Drexel University, Philadelphia (1994).



Experimental Conditions: P = 1 atm, Q = 1 LPM, 400ppm C4H10/3000ppm O2 / Ar balance

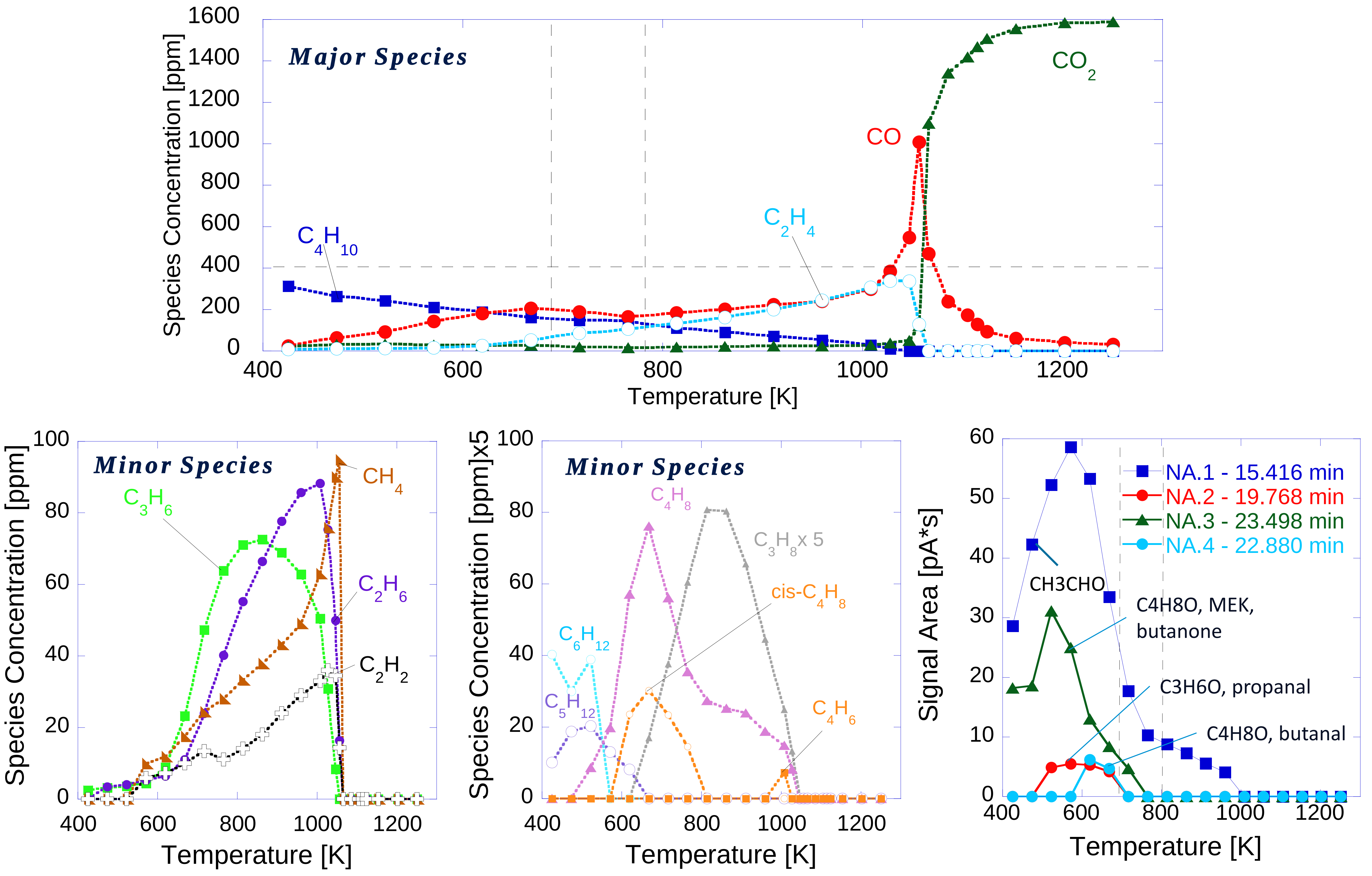




C_4H_{10} Plasma Oxidation

Experimental Conditions: P = 1 atm, Q = 1 LPM, 400ppm C4H10/3000ppm O2 / Ar balance

Plasma Conditions: 10 kV, ν = 1 kHz



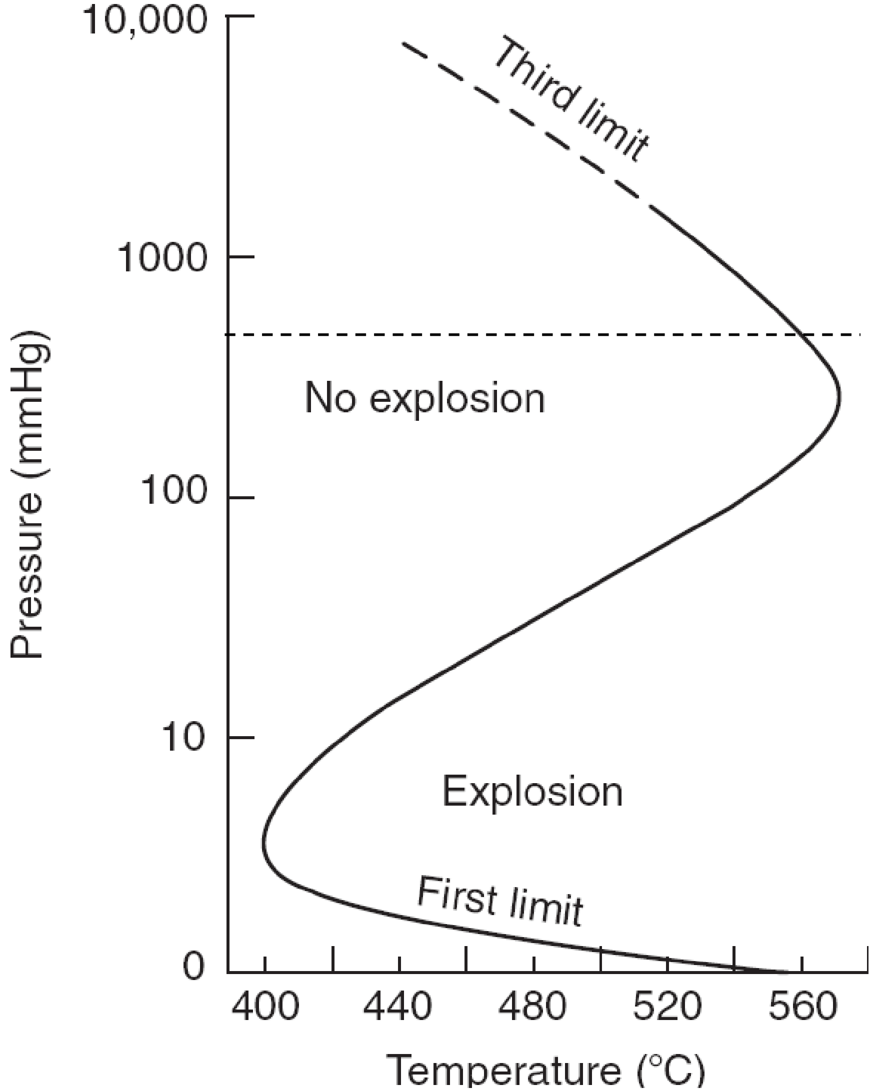
H_2 Plasma Assisted Oxidation

H_2 Thermal and Plasma Assisted Oxidation

Experimental Conditions: P = 1 atm, Q = 1 LPM, 2000ppm H2/3000ppm O2 / Ar balance

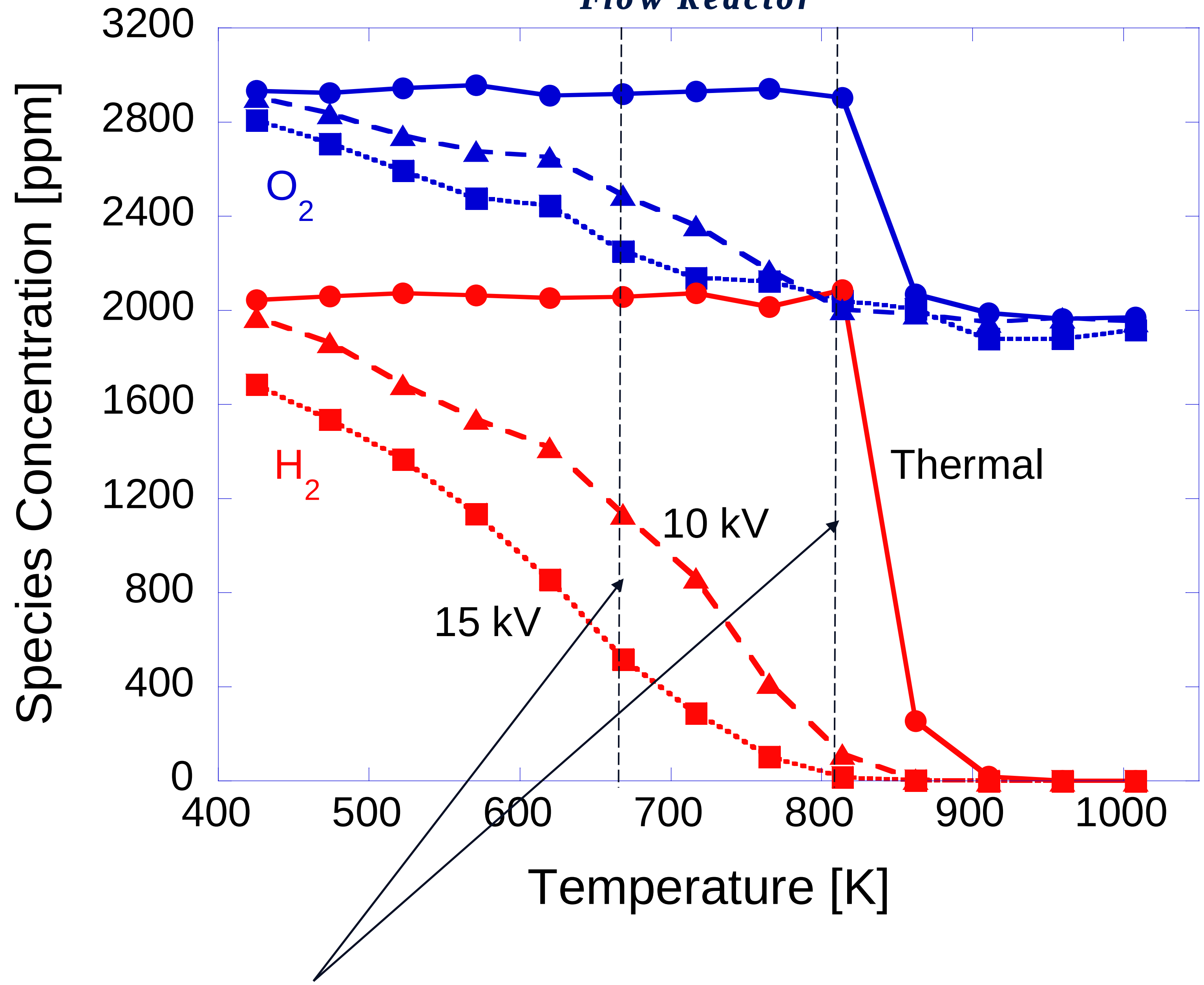
Plasma Conditions: 10kV and 15 kV, v = 1 kHz

H2/O2 Explosion Limits



I. Glassman and R.A. Yetter, **Combustion**, 4th Ed, Academic Press, 2008

Flow Reactor



OH LIF Experiments @ T =668K and T =814K

0D Kinetics Model with Sensitivity Analysis

SENKIN (CHEMKIN)

- Calculate $k(T)$
- Time integration
- Isothermal region modeled as constant T and P process
- Temperature ramps currently not included
- Plug flow assumption

$$k = AT^n \exp\left(-E_a / RT\right)$$

$$\frac{\partial N_i}{\partial t} = \omega_i$$

ZDPlasKin, a Boltzmann equation solver
(University of Toulouse, LAPLACE, 2008)

- Calculate rates for electron-impact reactions
- Electron impact cross sections are fed
- Calculate v_{drift}

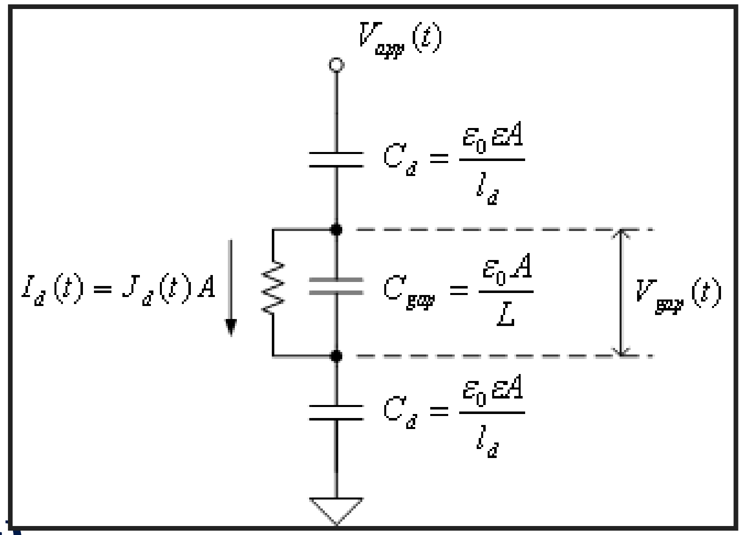
Circuit module

- Calculate E/N
- Assumed homogeneity across discharge planes

$$\frac{E}{N} = \frac{V_{\mathrm{gap}}(t)/L}{N}$$

$$J_{\mathrm{p}} = eN_{\mathrm{e}} v_{\mathrm{drift}}$$

$$\frac{\mathrm{d}V_{\mathrm{gap}}(t)}{\mathrm{d}t} = \left(1 + \frac{2l_{\mathrm{d}}}{\varepsilon \mathrm{L}}\right)^{-1} \left(\frac{\mathrm{d}V_{\mathrm{app}}(t)}{\mathrm{d}t} - \frac{2l_{\mathrm{d}}}{\varepsilon_0 \varepsilon} J_{\mathrm{p}}(t)\right)$$



Preliminary H2 Chemical Reaction Mechanism (39 species, 527 reactions)

Electron-impact reactions (Van Gaens and Bogaerts, 2013 and Kossyi et al., 1992)

Excitation, Deexcitation, Dissociation, Ionization, Recombination

Excited and ionized species (Van Gaens and Bogaerts, 2013, Kossyi et al., 1992 and Popov, 2011)

Ar/H/O atoms

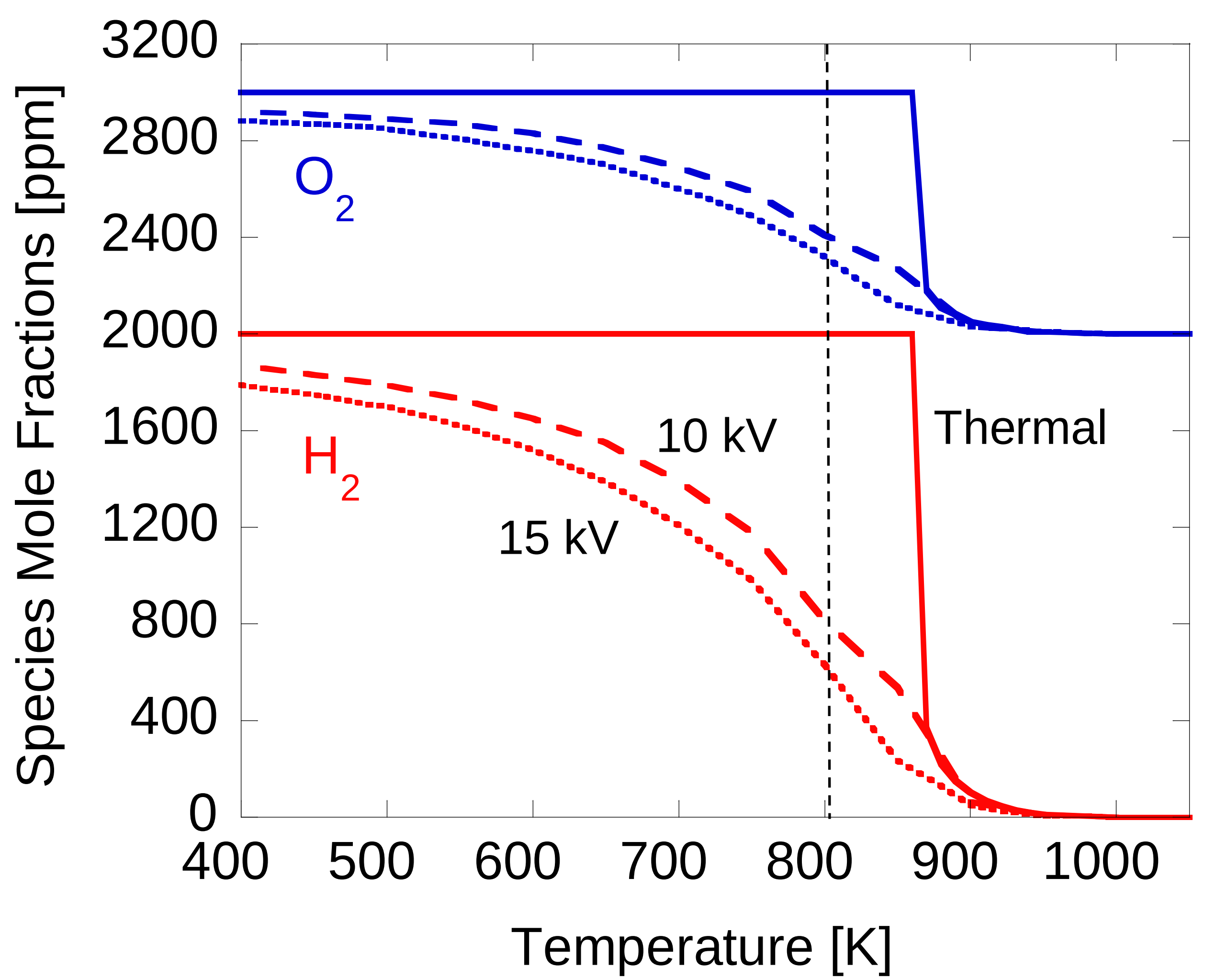
Neutral, ground state species

H_2/O_2 Mechanism (Popov, 2008), O_3 Chemistry (Bromly et al., 1996)

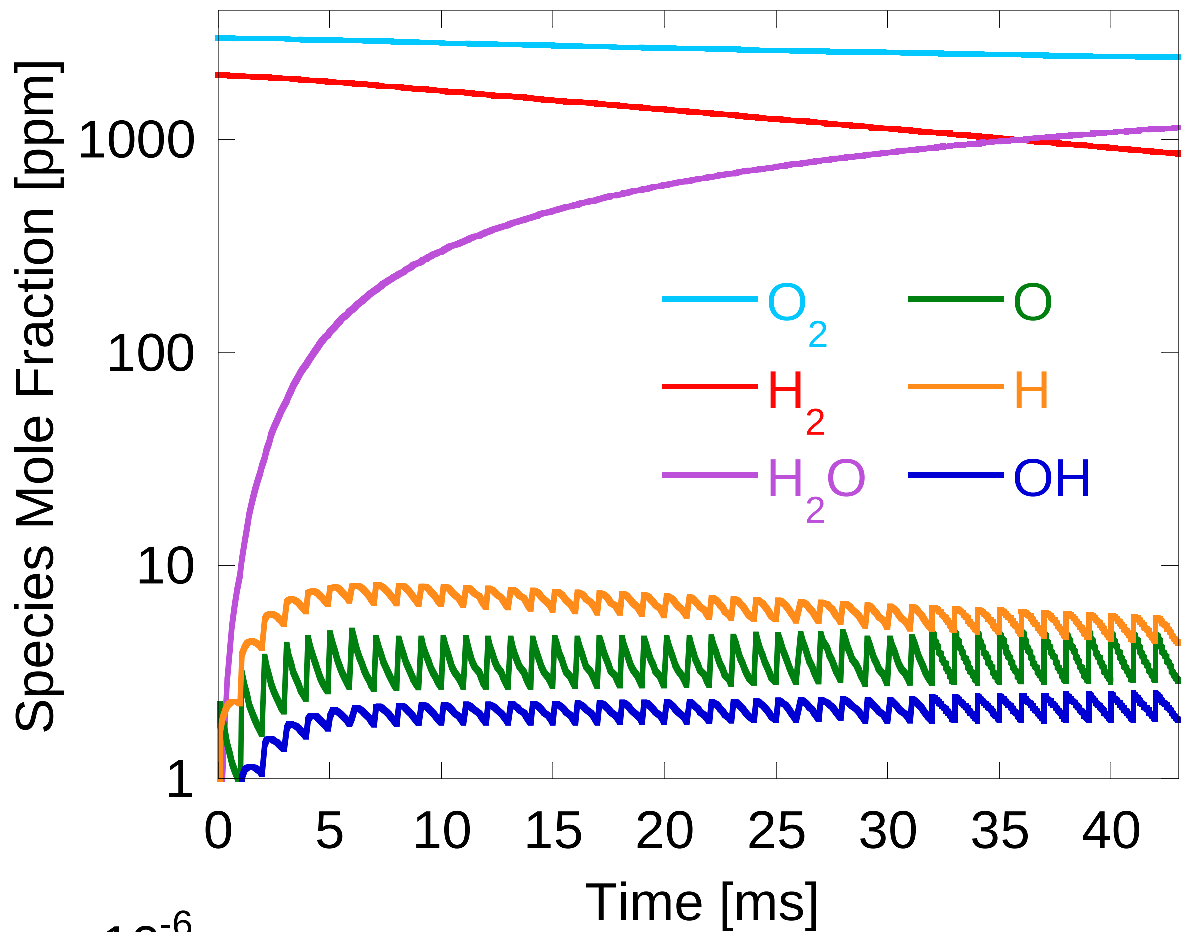
Wall quenching of positive ions

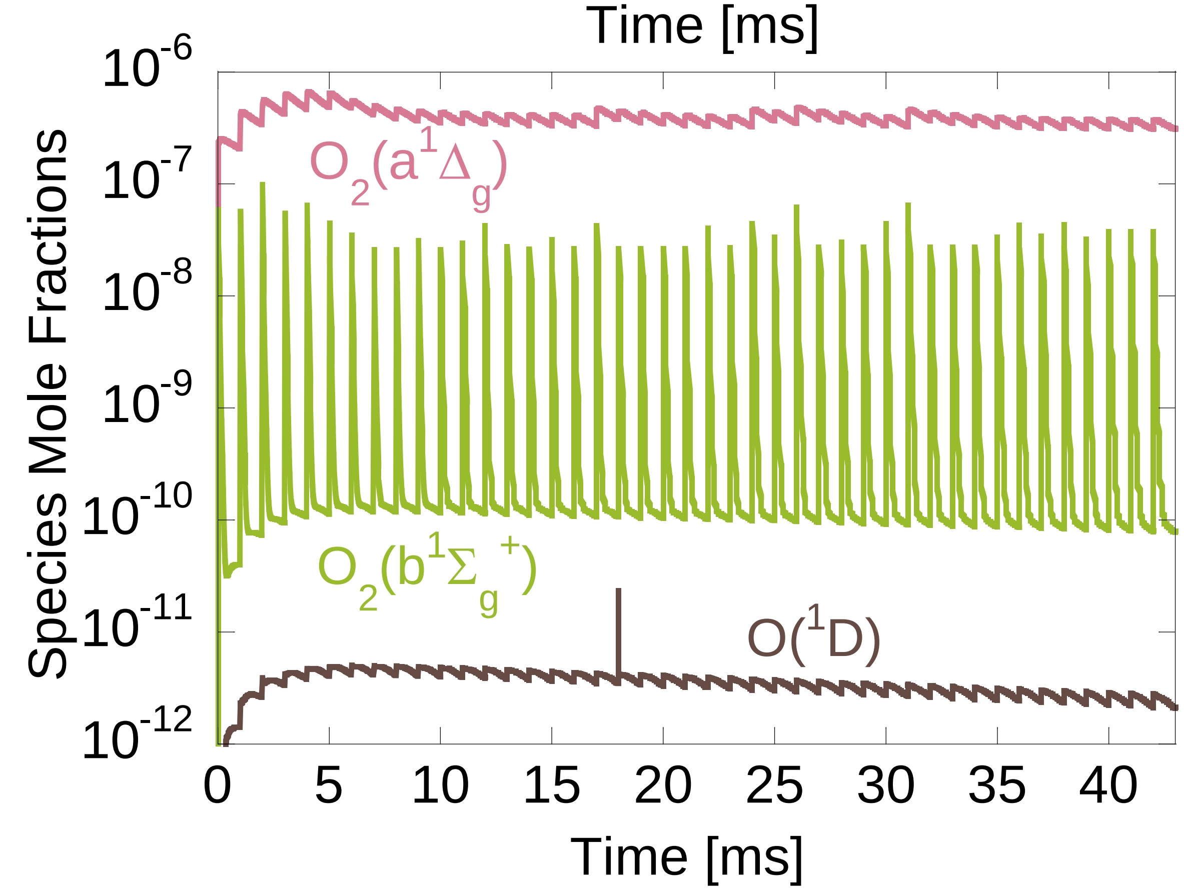
H_2 Oxidation (0D Kinetic Model)

Conditions: P = 1 atm, Q = 1 LPM, 2000ppm H2/3000ppm O2 / Ar balance
Plasma Conditions: 10kV and 15 kV, ν = 1 kHz

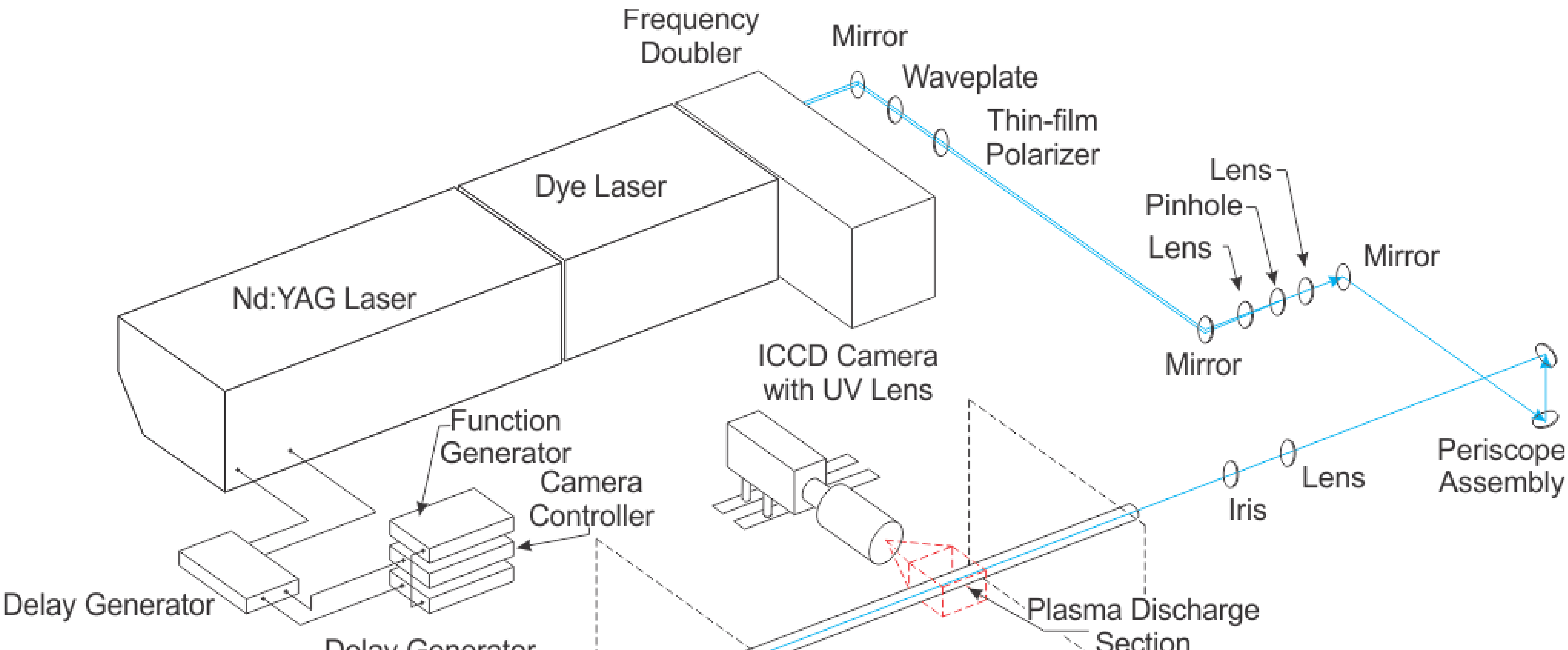


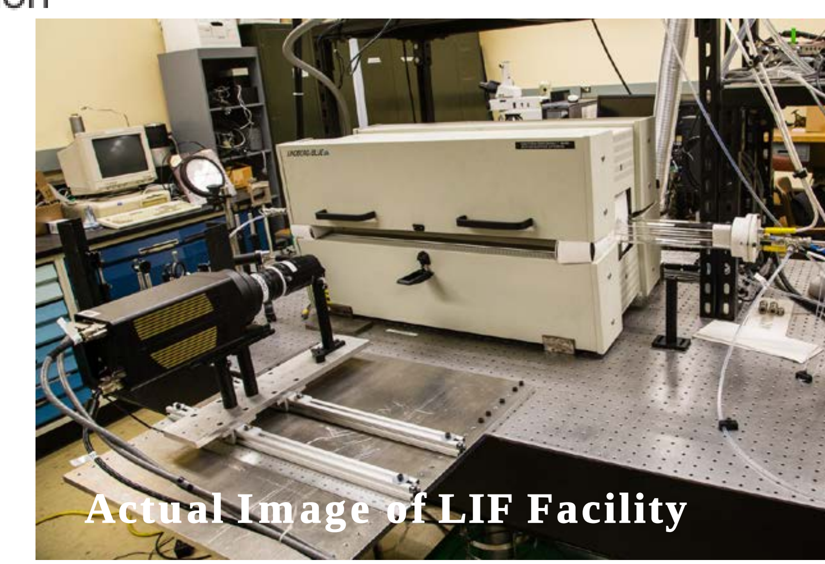
T = 800K, 10kV, v = 1 kHz
Electrode Region





OH LIF Experimental Extension

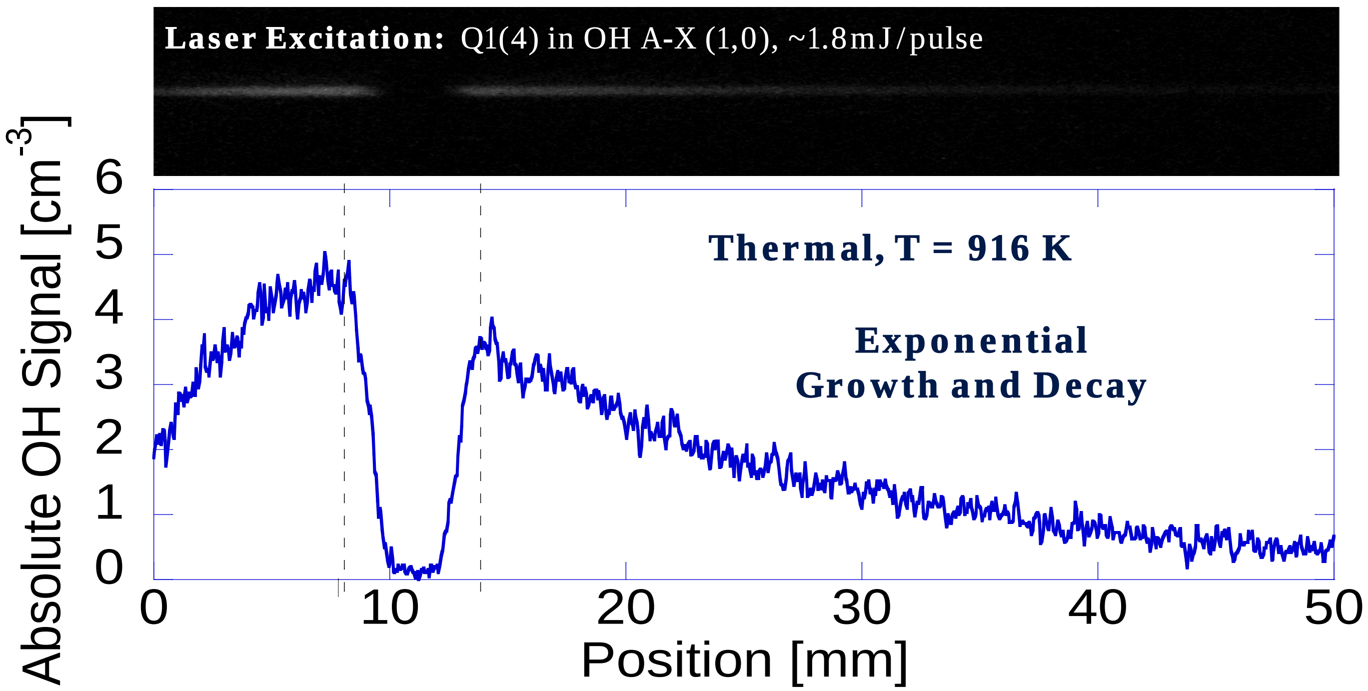


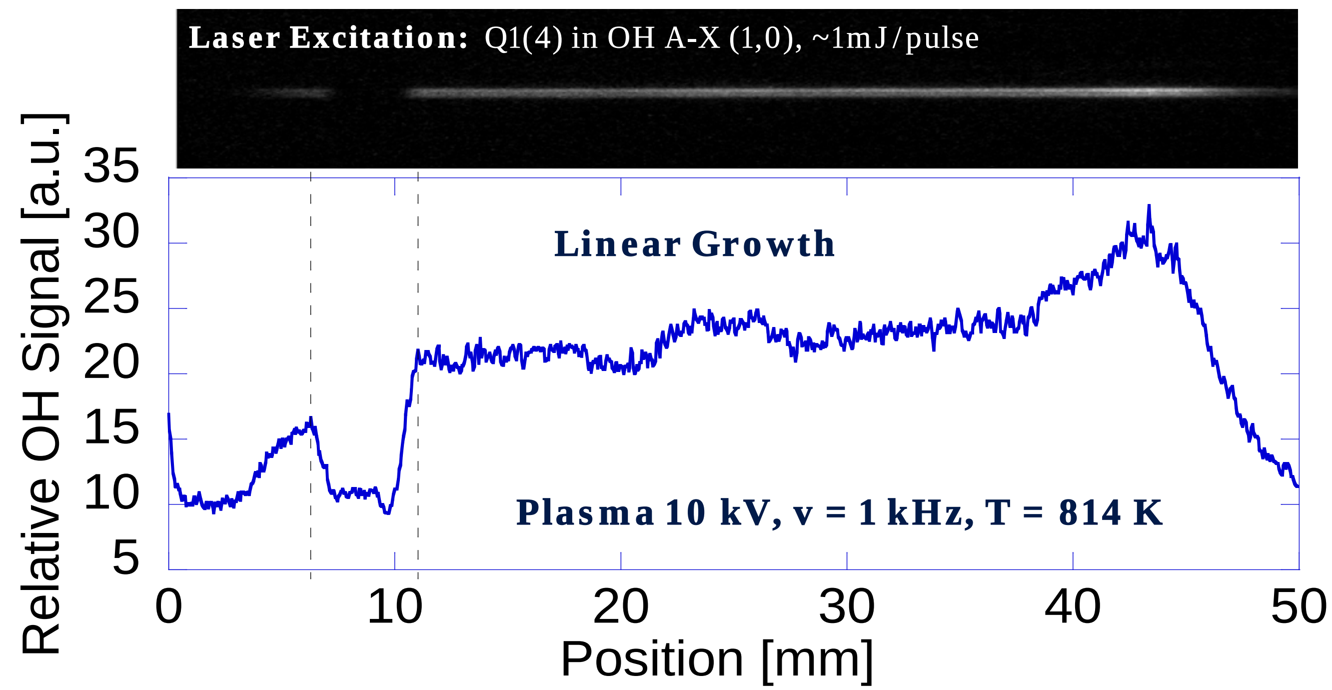

Actual Image of LIF Facility

- *in-situ* OH LIF implemented into existing flow reactor experiment in collaboration with OSU group (Zhiyao Yin and Kraig Frederickson)
- ICCD camera mounted on **translational stage** to obtain measurements along the length of the reaction zone

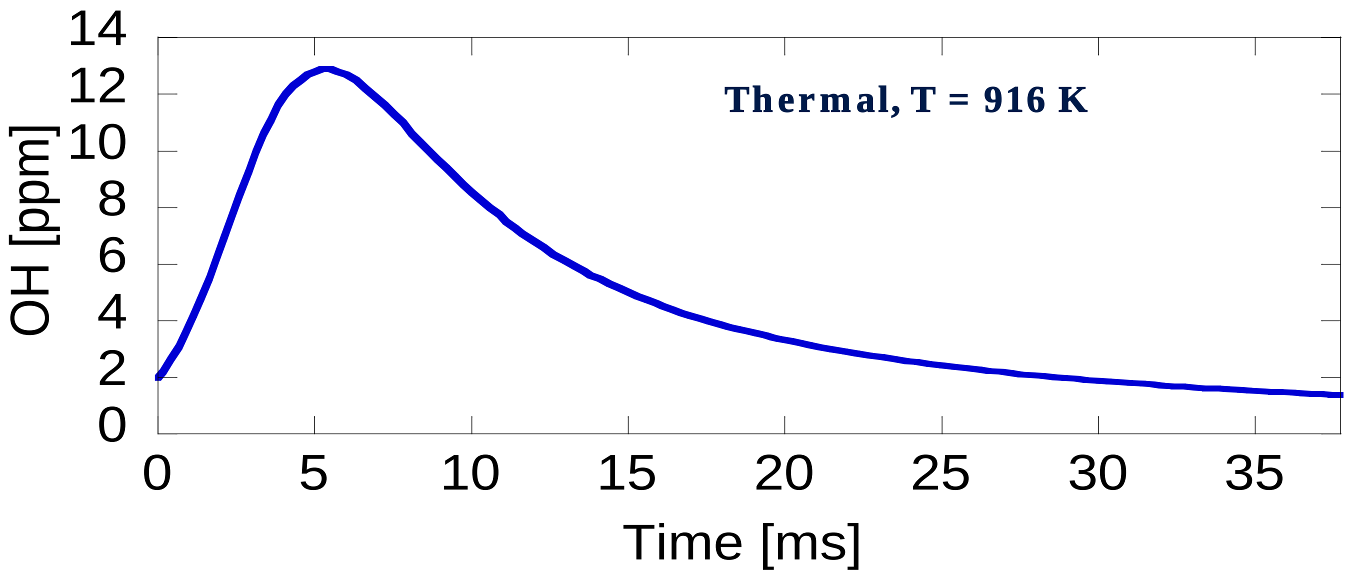
OH LIF Experiments

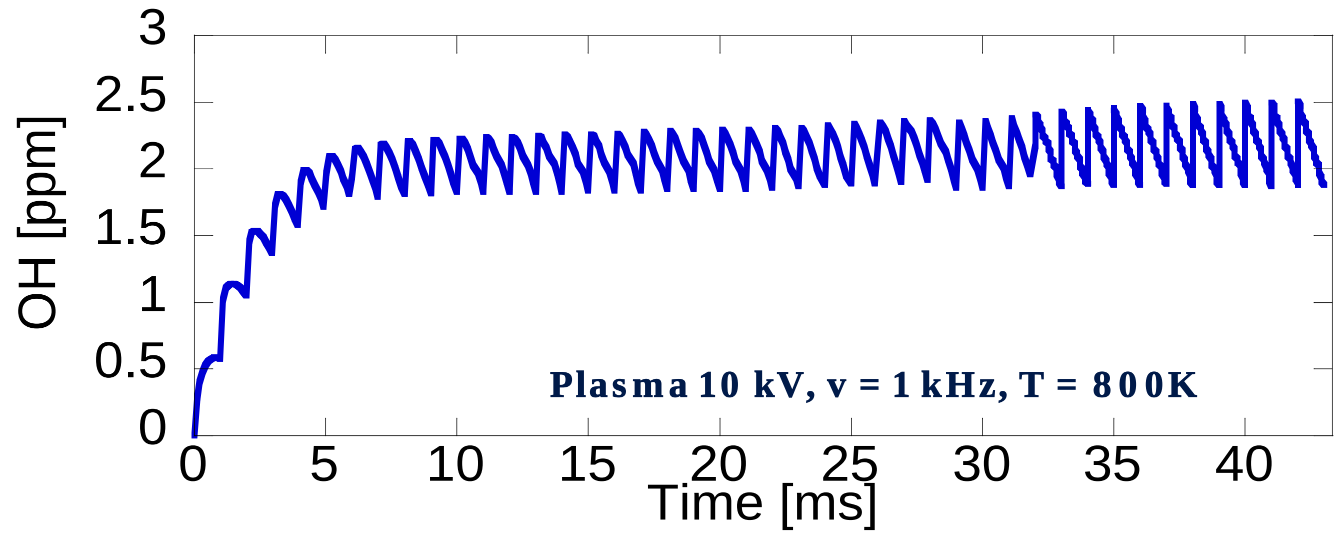
Experimental Conditions: P = 1 atm, Q = 1 LPM, 2000 ppm H2/3000ppm O2 / Ar balance
600 Shots On- ICCD Accumulations





OH 0D Plasma Chemical Kinetics Code



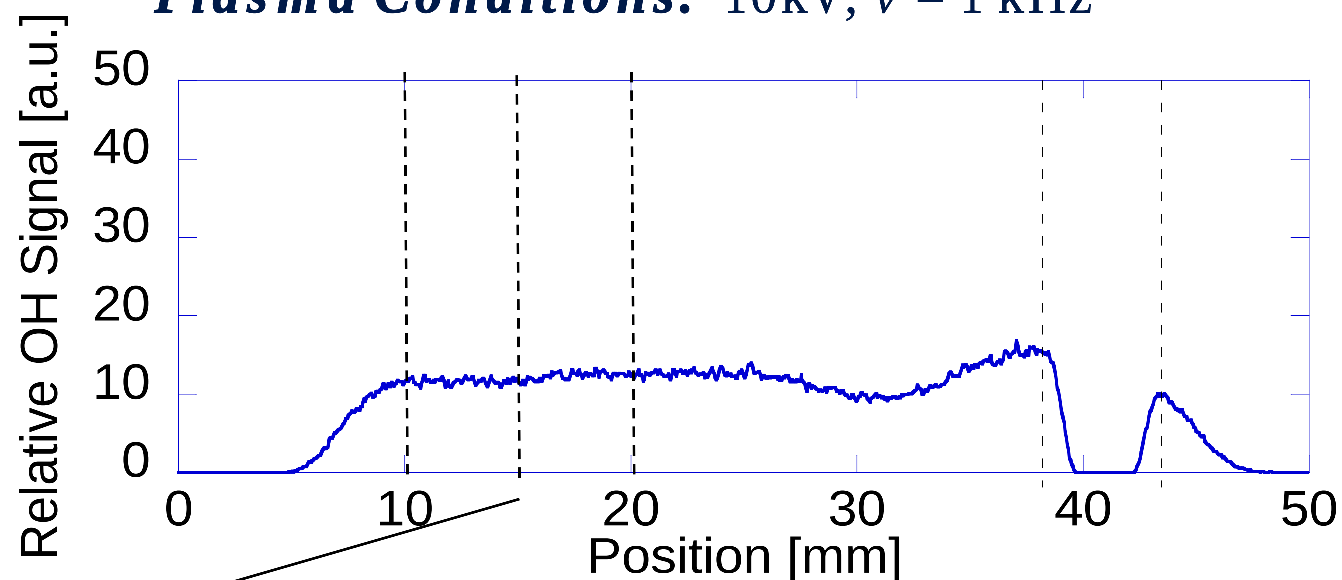


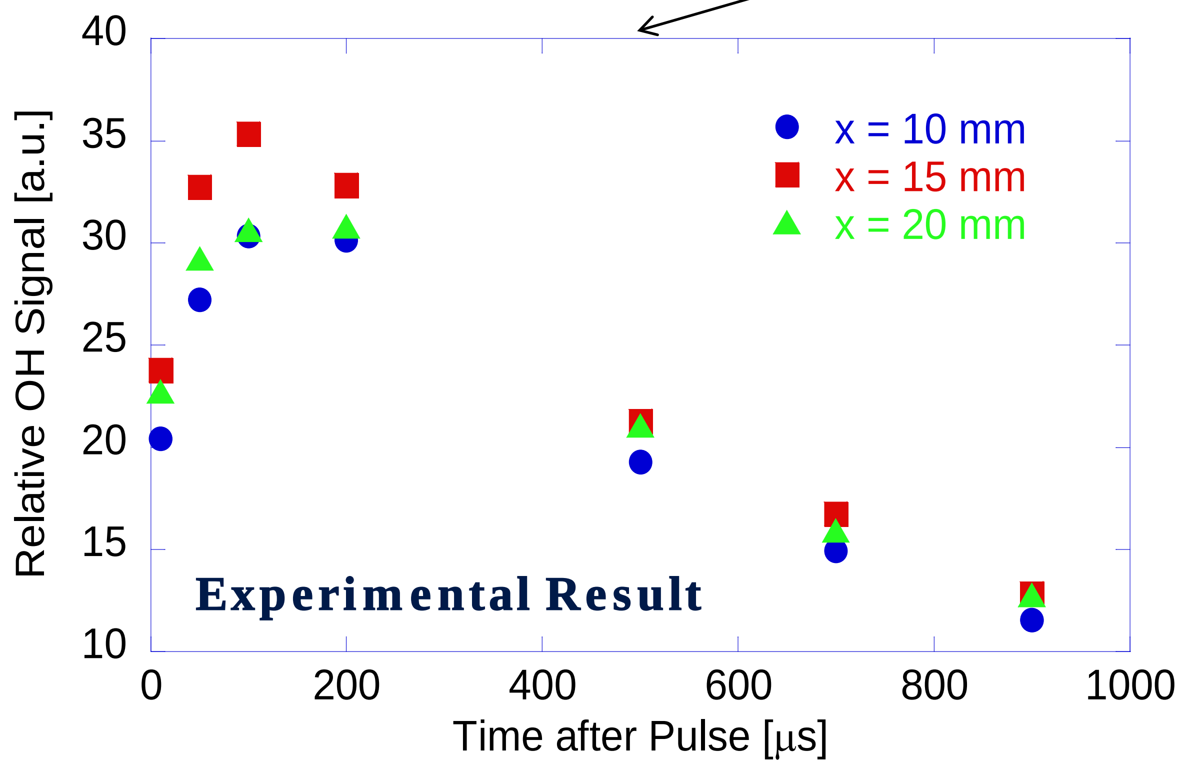
Transient Behavior of OH at a Specific Reactor Location

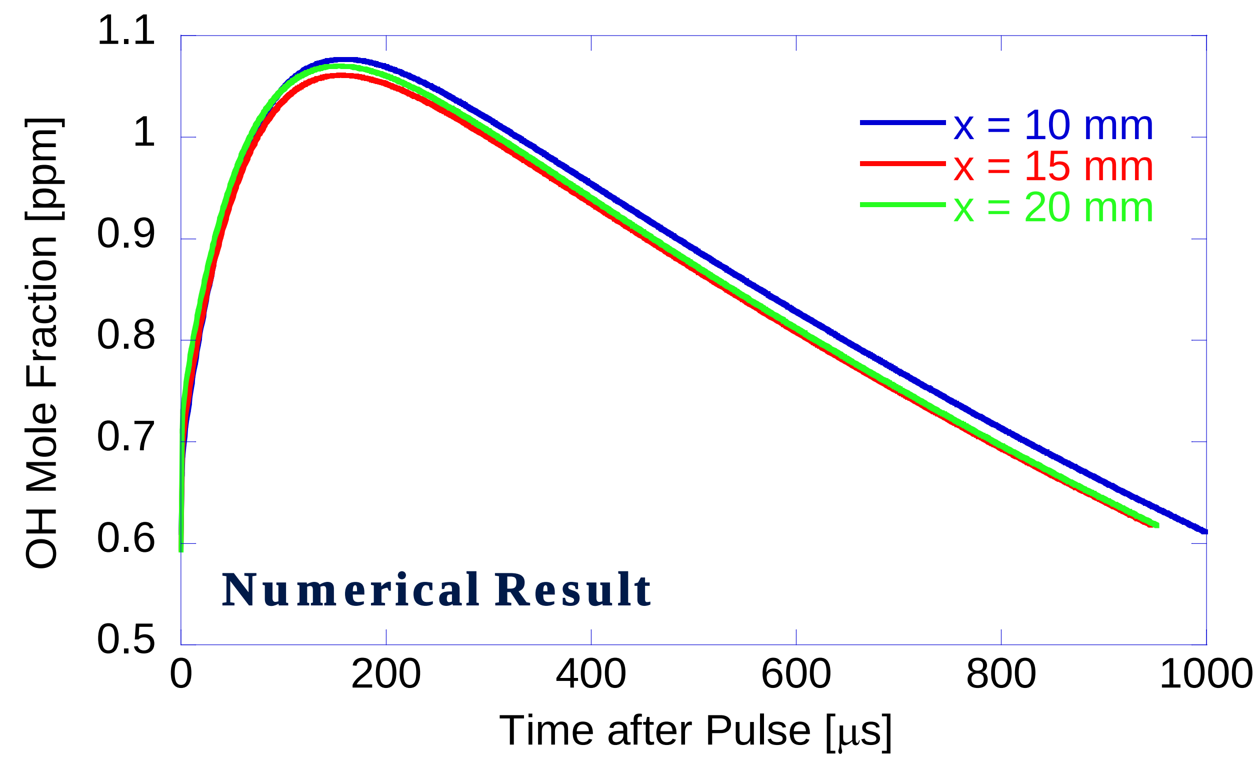
Conditions: P = 1 atm, T=668 K, Q = 1 LPM, 2000ppm H2/3000ppm O2 / Ar by balance

Plasma Conditions: 10kV, ν = 1 kHz

Example of Experimental OH Signal @ t = 900 μs







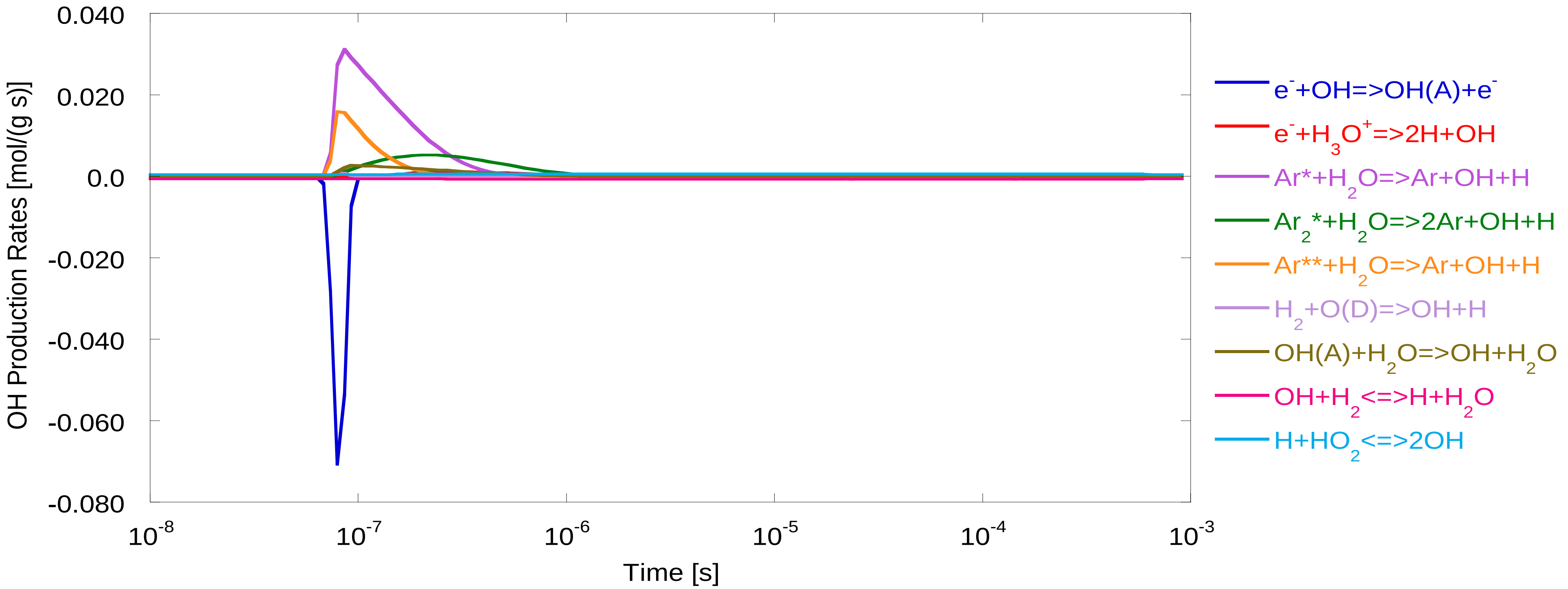
OH Production and Sensitivity

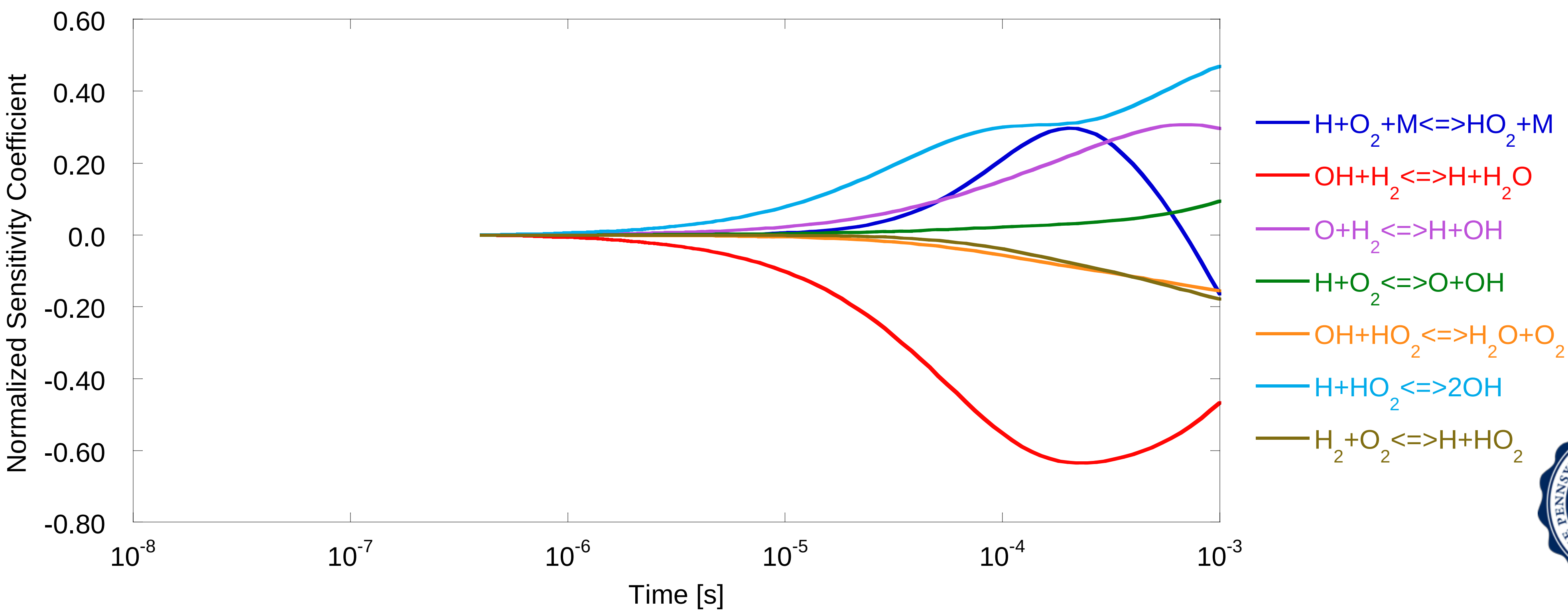
Conditions: P = 1 atm, T=800 K, Q = 1 LPM, 2000ppm H2/3000ppm O2 / Ar by balance

Plasma Conditions: 10kV, ν = 1 kHz

Pulse #22

Discharge at 77 ns, V_{peak} at 100 ns





Summary

- Alkane (C1-C7) oxidation has been studied with and without plasma enhancement:
  - Fuel consumption across T = 420 – 1250 K has been significantly enhanced for all fuels, the plasma also lowers the temperature for complete fuel consumption (*the temperature for onset of high temperature ignition*)
  - Experimental results indicate an extension of RO2 low temperature (cool flame) chemistry to lower pressures (*identification/quantification of select species will be improved i.e., aldehydes)*
- H2 oxidation has been studied with and without plasma enhancement:
  - H2 and O2 consumption measured *ex-situ* GC analysis, modifications to the current facility have been made to perform *in-situ* OH LIF measurements in conjunction with OSU group
  - Zero-D plasma kinetic model has been developed based on SENKIN to derive detailed sensitivity analysis
  - Qualitative comparisons of model and experiment indicate current plasma assisted combustion models for hydrogen oxidation perform well.
- Experiment and model analysis of low temperature C2H4/N2/O2/Ar mixtures suggest intermediate formation of nitromethane. Formation of such nitro and nitrate compounds at low temperatures may be a means to enhance ignition.

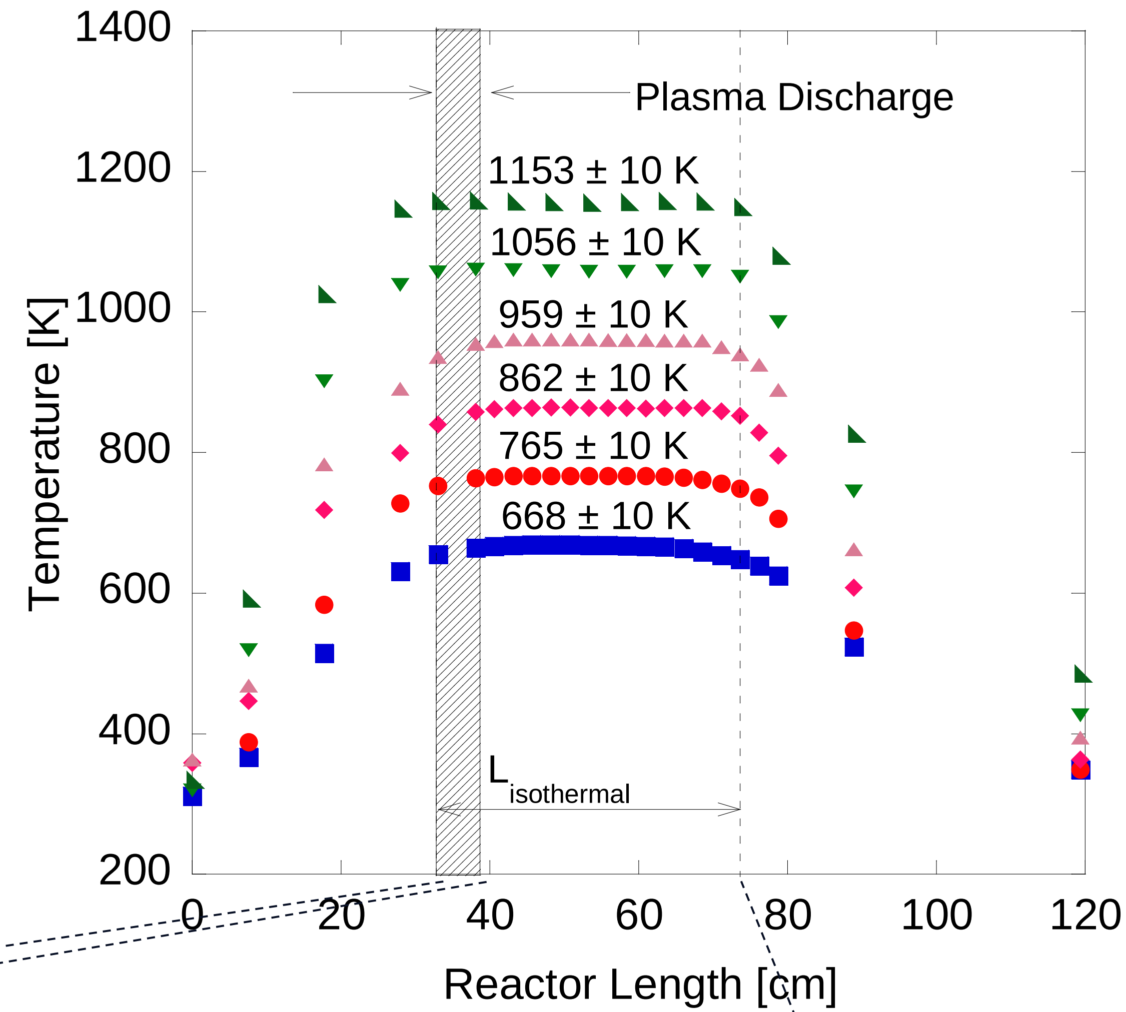
Team Interactions/Collaborations

- Collaborative research with Ohio State on plasma kinetic modeling of PSU pyrolysis and oxidation experiments.
- Collaborative research with Ohio State on *in-situ* OH LIF measurements.
- Plasma Assisted Combustion MURI Kinetics Working Group

Secondary Slides

0D Kinetics Model

- **Isothermal** region modeled as *constant* **Temperature** and **Pressure** process
- Temperature ramps ignored (*to be implemented in the future*)
- Assumed **homogeneity** across discharge planes
- **Plug flow** assumption
- Convert **distance to time** based on experimental flow rate



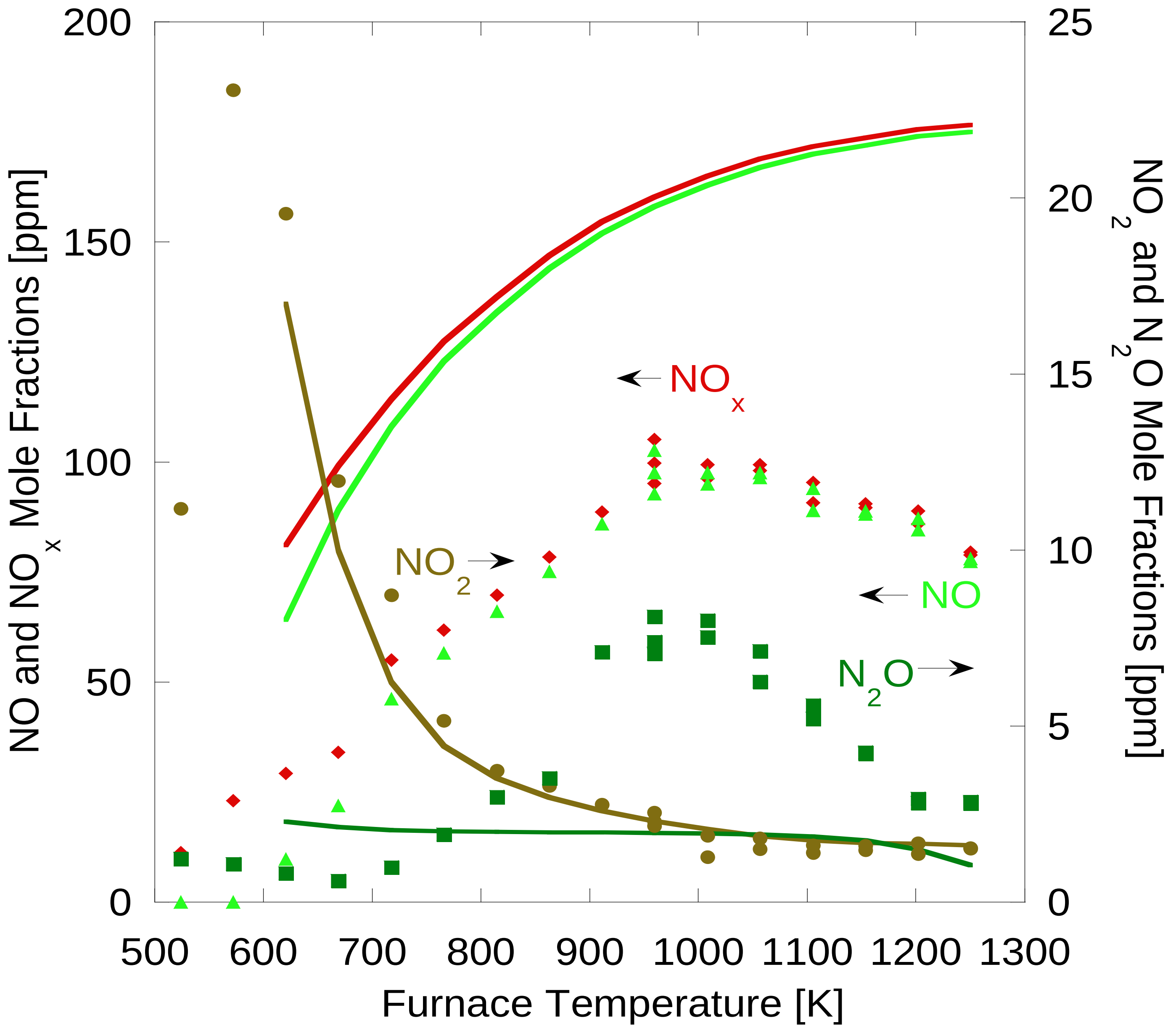
Preliminary Chemical Reaction Mechanism
114 species, 1461 reactions

- Electron-impact reactions (Mainly from Van Gaens and Bogaerts, 2013 and Kossyi *et al*., 1992)
- Excitation
- Deexcitation
- Dissociation
- Ionization
- Recombination
- Excited and ionized species (Mainly from Van Gaens and Bogaerts, 2013 and Kossyi *et al*., 1992)
- Ar/H/N/O atoms
- Interactions of hydrocarbons with excited species
- Neutral, ground state species
- Hydrocarbon oxidation (Bromly *et al*., 1996)
- NO_x Mechanisms
  - *Zel'dovich (Bromly et al., 1996)*
  - *Prompt, N_2O and NNH (GRI-Mech 3.0)*

Plasma Effects on NO_x Formation at Various Temperatures

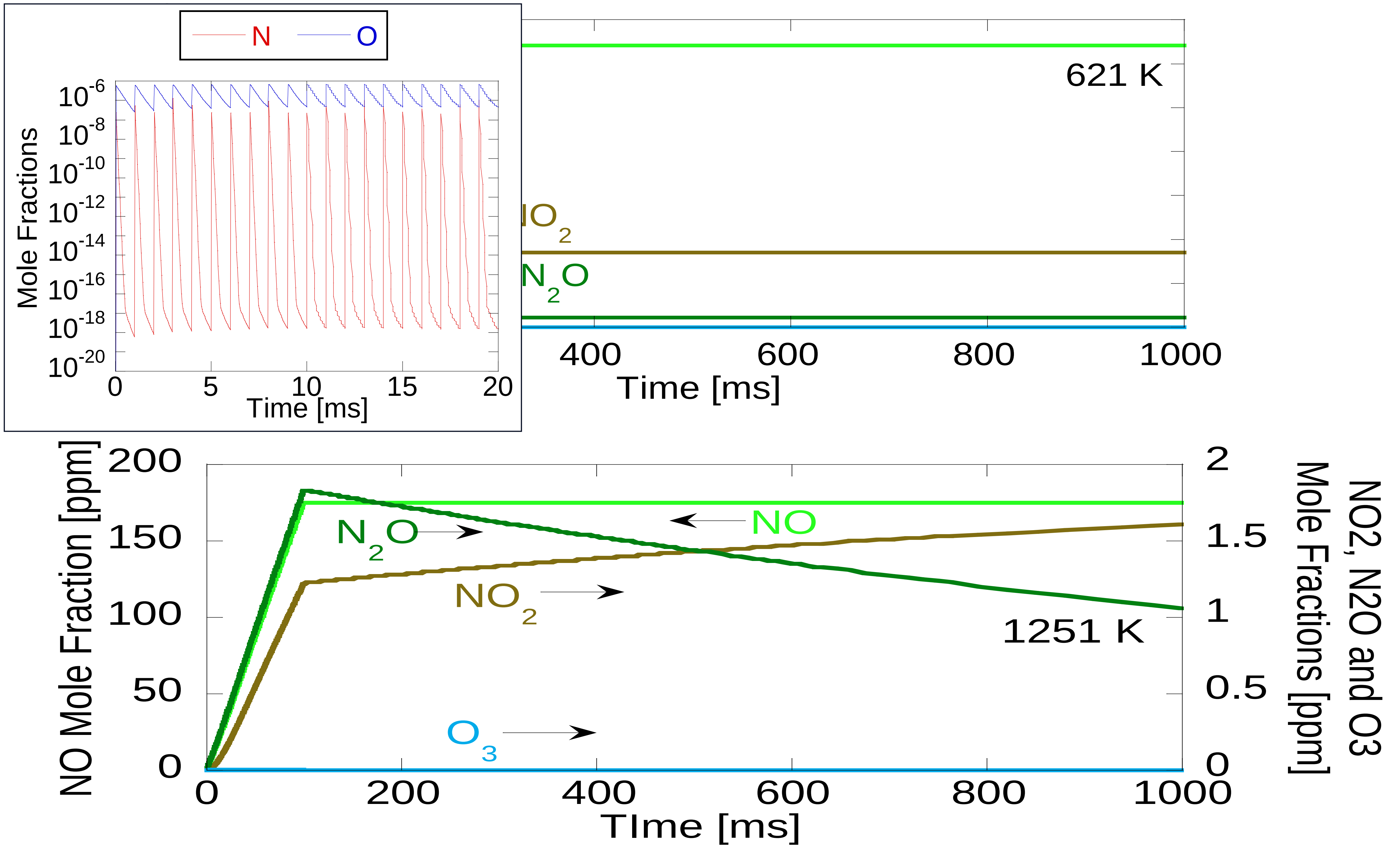
50% Ar / 50% Air
p = 1 atm
Plasma residence time, τ_{plasma} = 100 ms
V_{peak} = 15 kV
Repetition rate, f = 1 kHz

Discrete points: experimental results
Solid lines: numerical results



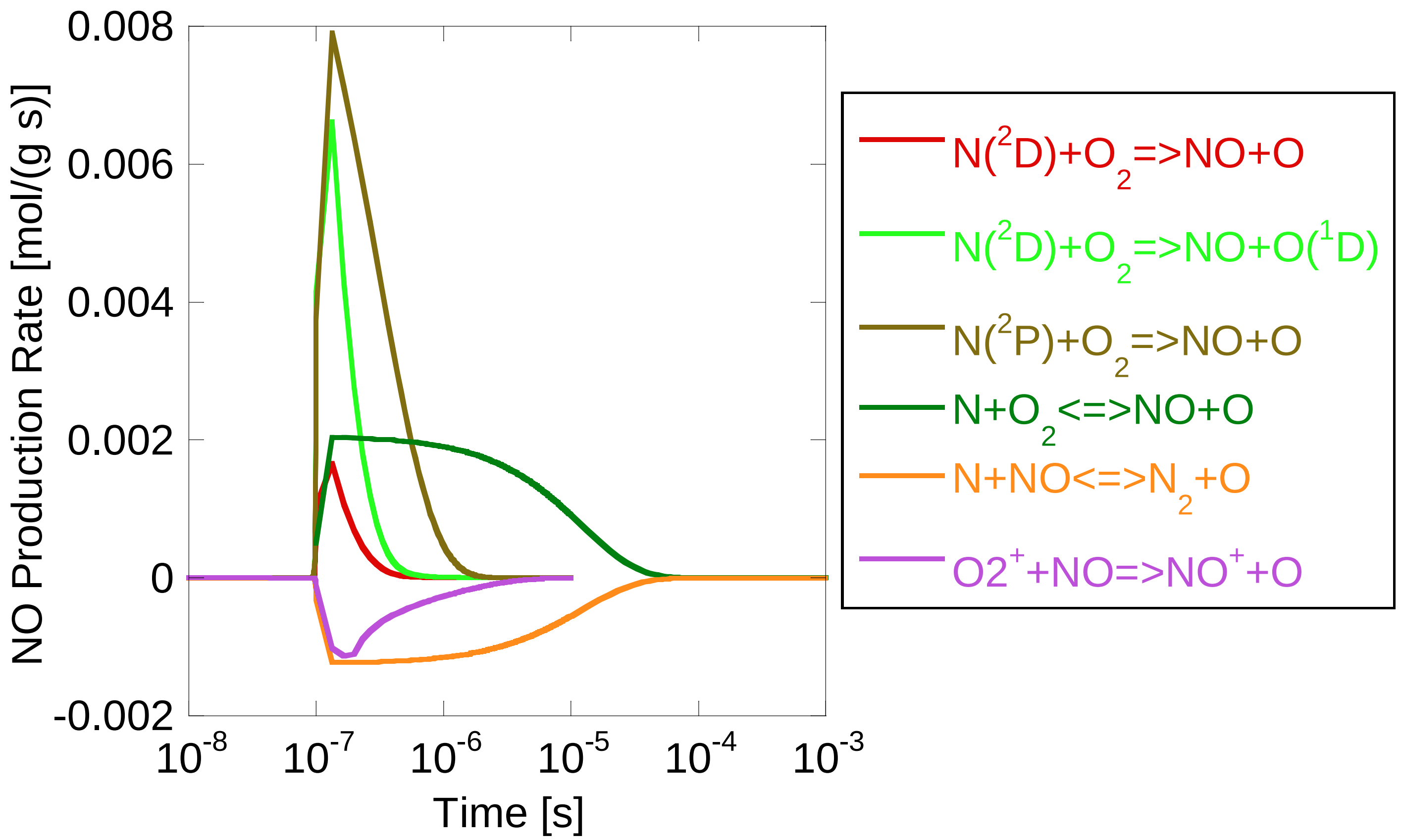
- N atom production results from electron impact dissociation of N_2.
- NO_x forms by $N+O_2 \rightarrow NO+O$. NO increases with temperature because of more N atom formation.
- Model overpredicts NO by a factor of 2. Most likely due to overprediction of N atom formation by electron impact dissociation. Total NO_x measured also decreases for temperatures above 1050 K.
- Higher portion of NO_2 at low temperatures due to the reaction $NO+O_3 \rightarrow NO_2+O_2$. O_3 decreases with temperature, so does NO_2.

Plasma Effects on NO_x Formation at Various Temperatures: Simulated Species History

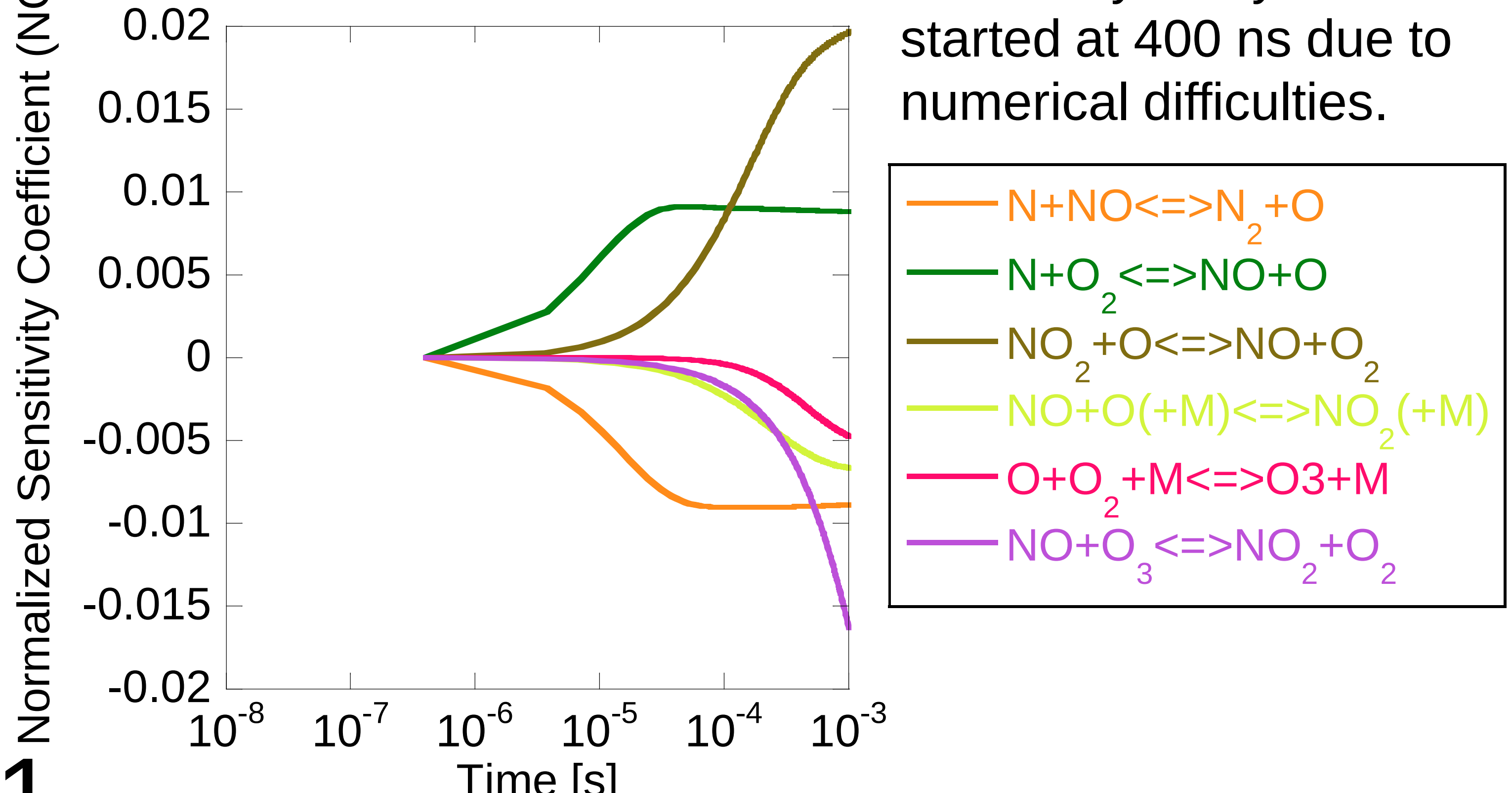


NO Analyses (Production rate and Sensitivity)

Pulse #100 of 620.7K case



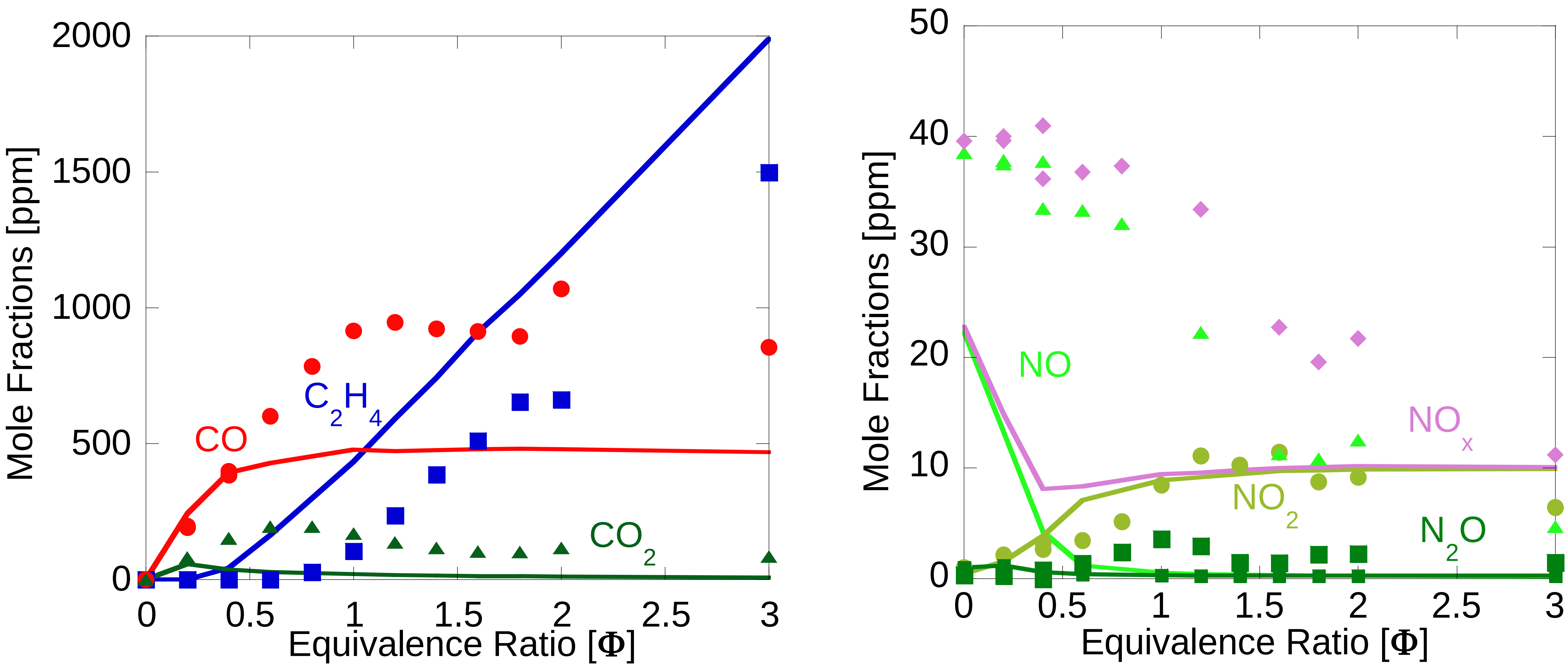
Sensitivity analysis started at 400 ns due to numerical difficulties.



- NO production due to $N+O_2=NO+O$. Reverse reaction $N+NO=N_2+O$ present because NO already built up.
- After 400 ns, NO is not sensitive to excited or ionized species any more.
- Sensitivity analysis shows three important mechanisms in the after glow: Zel'dovich thermal NO_x mechanism, $NO \leftrightarrow NO_2$ cycle by O atoms, and oxidation to NO_2 by O_3.

Plasma Effects on NO_x Formation at Various Equivalence Ratios

9600 ppm N_2 / 2400 ppm O_2 / (800Φ) ppm C_2H_4 /Balance Ar
1 atm, 814 K, τ_{plasma} = 70 ms, V_{peak} = 15 kV, f = 1 kHz



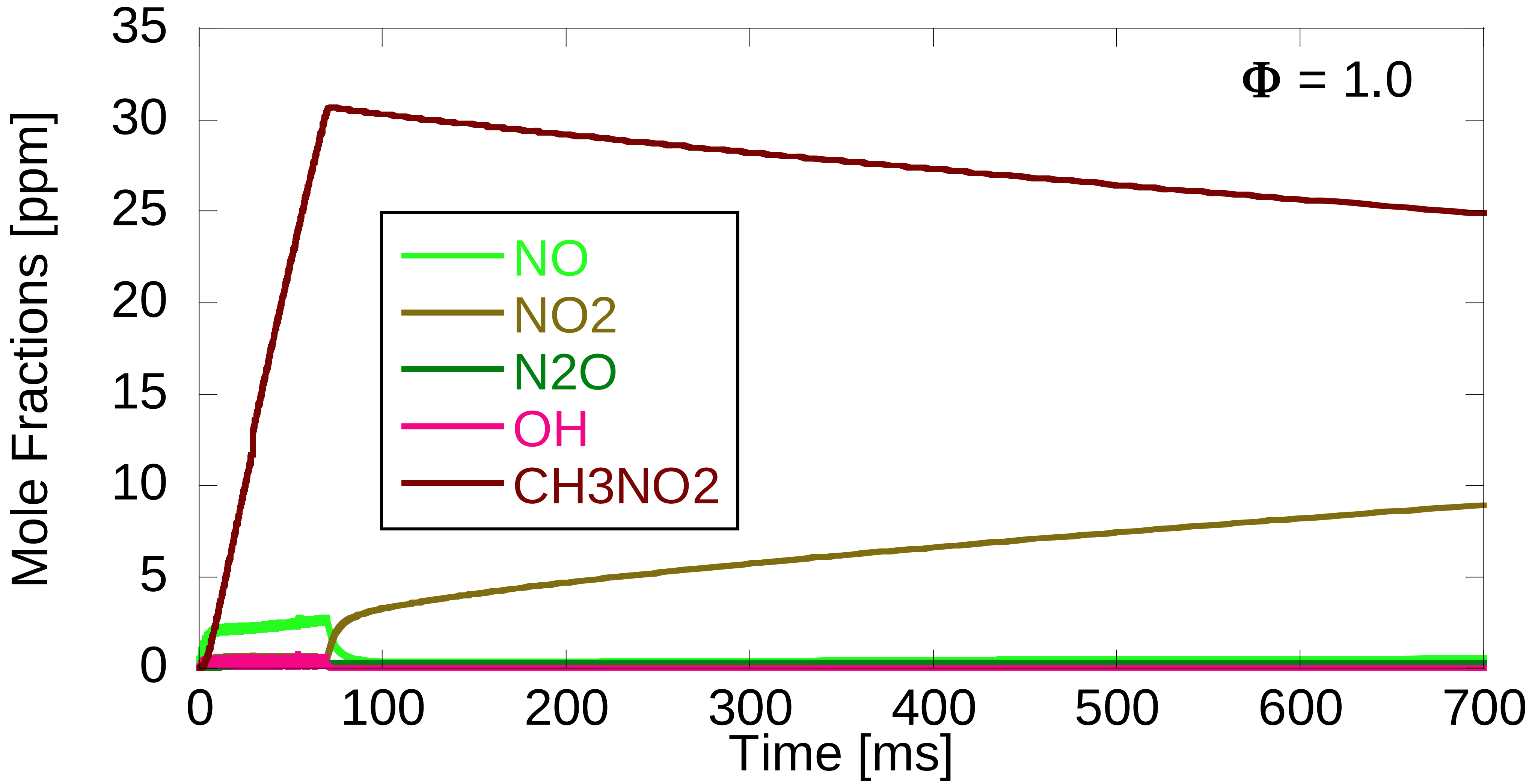
Experimental results

- For Φ>0.5, NO_x formation decreases with Φ.
- Reaction rate constant of dissociative quenching of Ar* with fuel ($Ar^*+C_2H_4 \rightarrow Ar+C_2H_3+H$ or $Ar^*+C_2H_4 \rightarrow Ar+C_2H_2+H_2$) is ≈10x faster than the one with N_2 ($Ar^*+N_2 \rightarrow Ar+2N$).

Numerical results

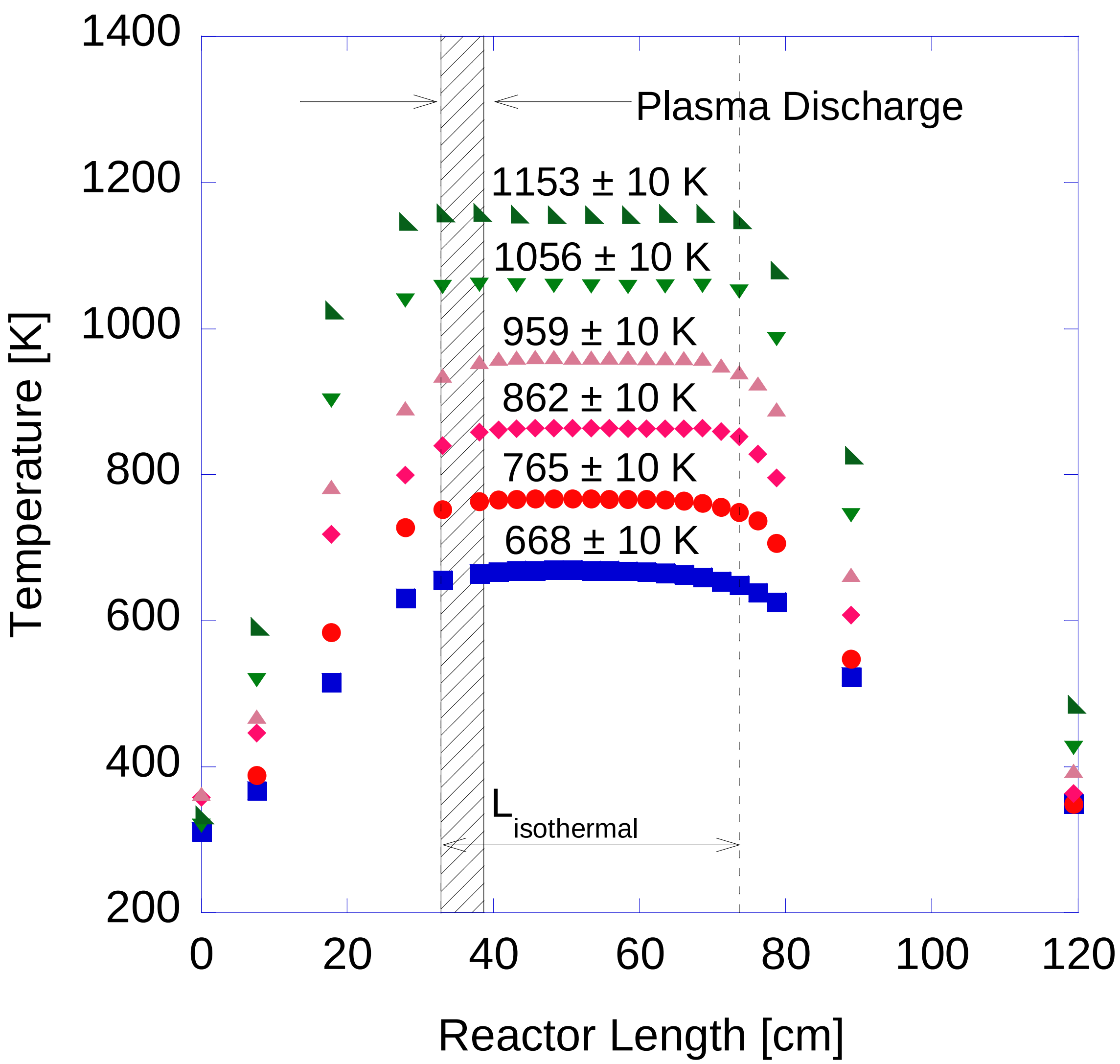
- Large discrepancies between experimental and numerical results. The trends of C-containing species profiles are qualitatively similar, but the matching of N-containing species are miserable.

What is happening in the code?

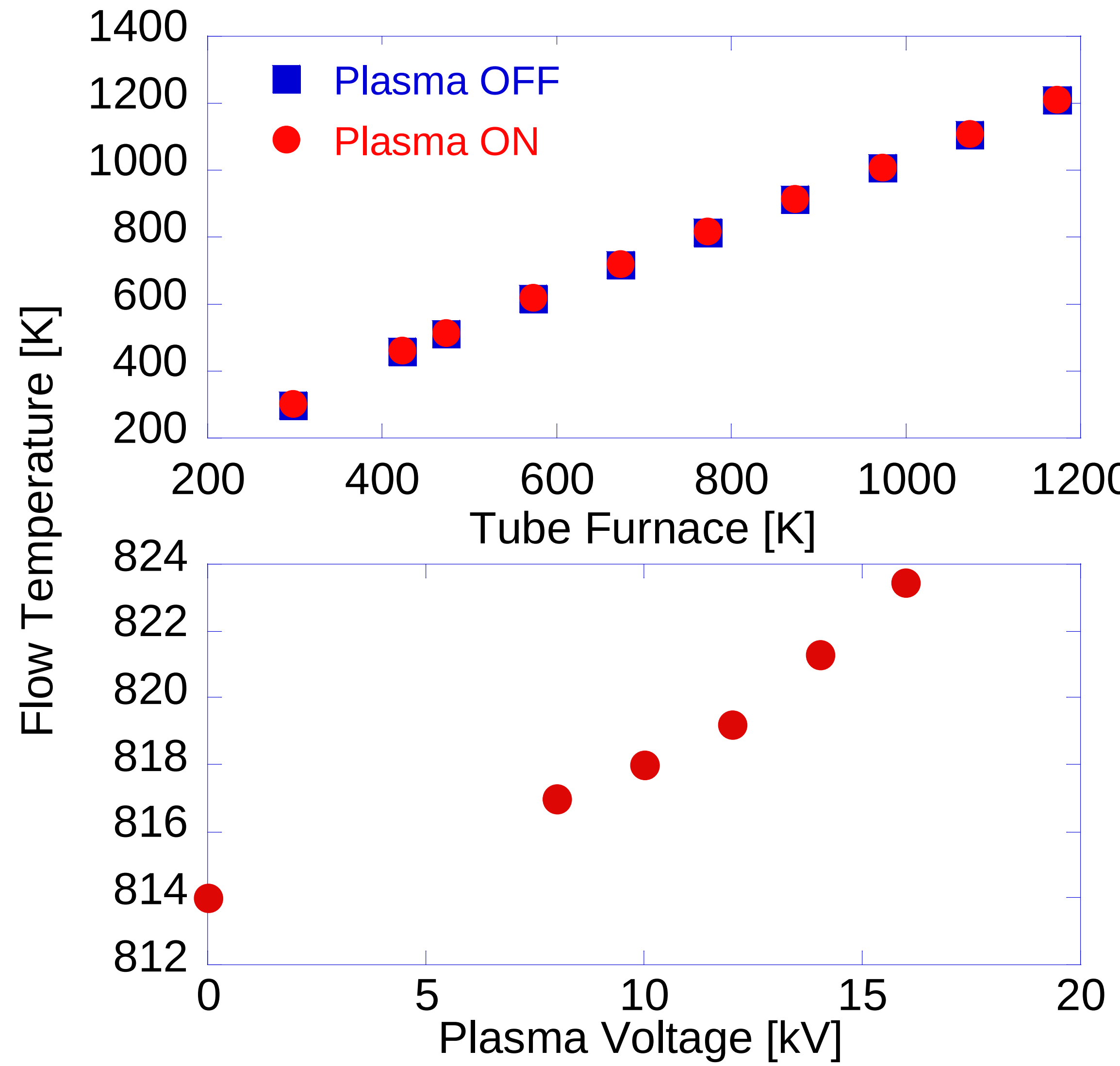


- NO forms in plasma region, and quickly converted to NO_2 by $NO+HO_2$ at the exit of plasma region.
- Large amount of nitromethane (CH_3NO_2) forms within the plasma region, by $CH_3+NO_2(+M)=CH_3NO_2(+M)$. Downstream, CH_3NO_2 then decomposes.
- Current model missing many secondary reactions of CH_3NO_2 decomposition.

Operating Conditions - Temperature



Reactor Temperature Profile
@ 1 LPM of Ar



1 LPM of Ar with
V= 10kV
ν = 1 kHz

1 LPM of Ar
with 3 sccm O2
ν = 1 kHz

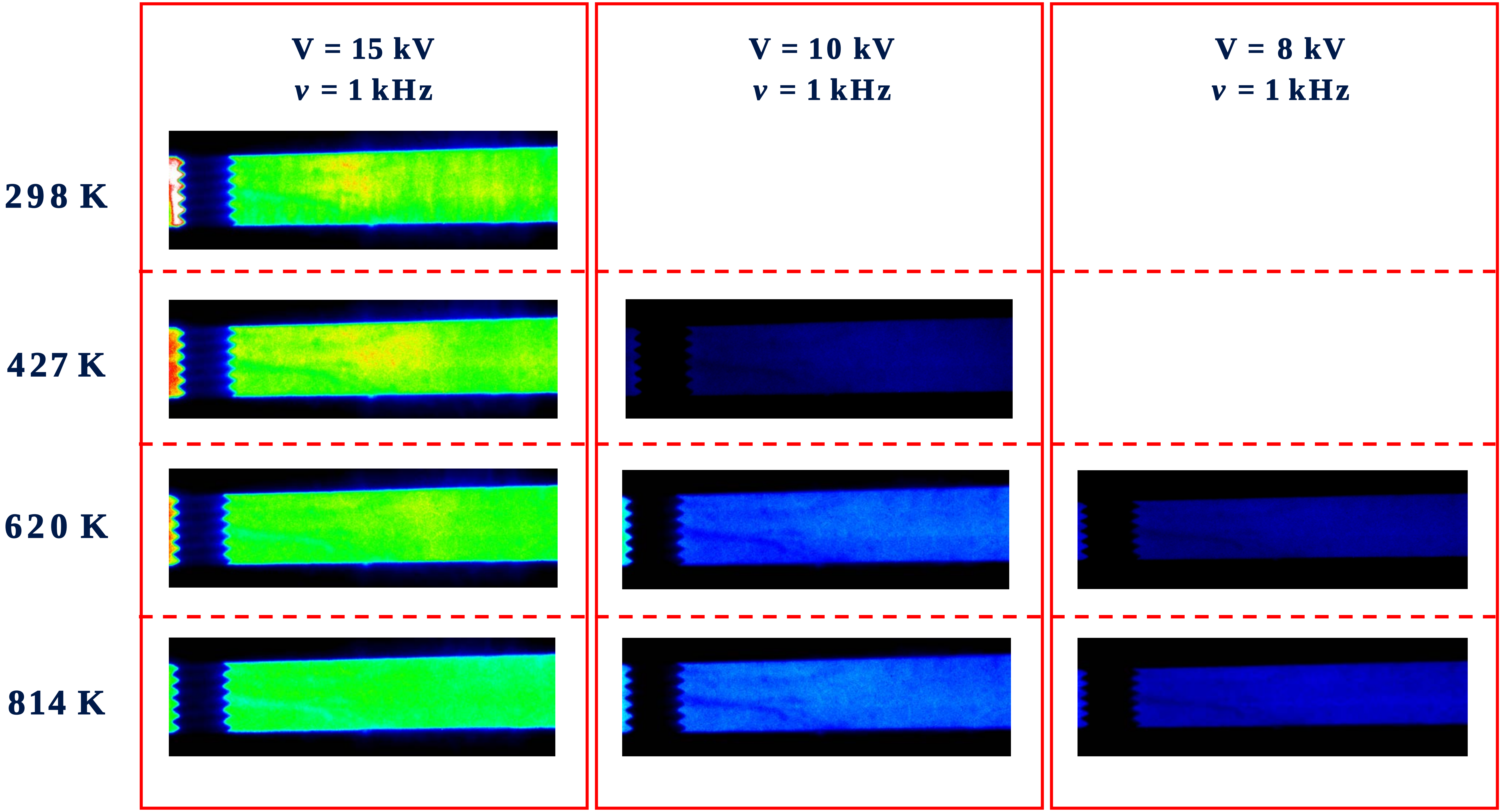
Effect of Plasma Discharge on Temperature

- The measured temperature profiles are used to establish an empirical correlation with the tube furnace temperature over a range of 350 K < T_{iso} < 1250 K. The correlation is used for kinetic modeling.
- Initial experiments verify that heating from the plasma discharge is negligible in the diluent flow
- In Ar/O2 flow, ~ 10 K increase in gas temperature observed at the maximum voltage setting for a given reactor temperature of 814K

Operating Conditions - Plasma Discharge

Experimental Conditions: P = 1 atm, Q = 1 LPM Ar, Gate = 10 μsec

ICCD Multiple Images (average of 50)



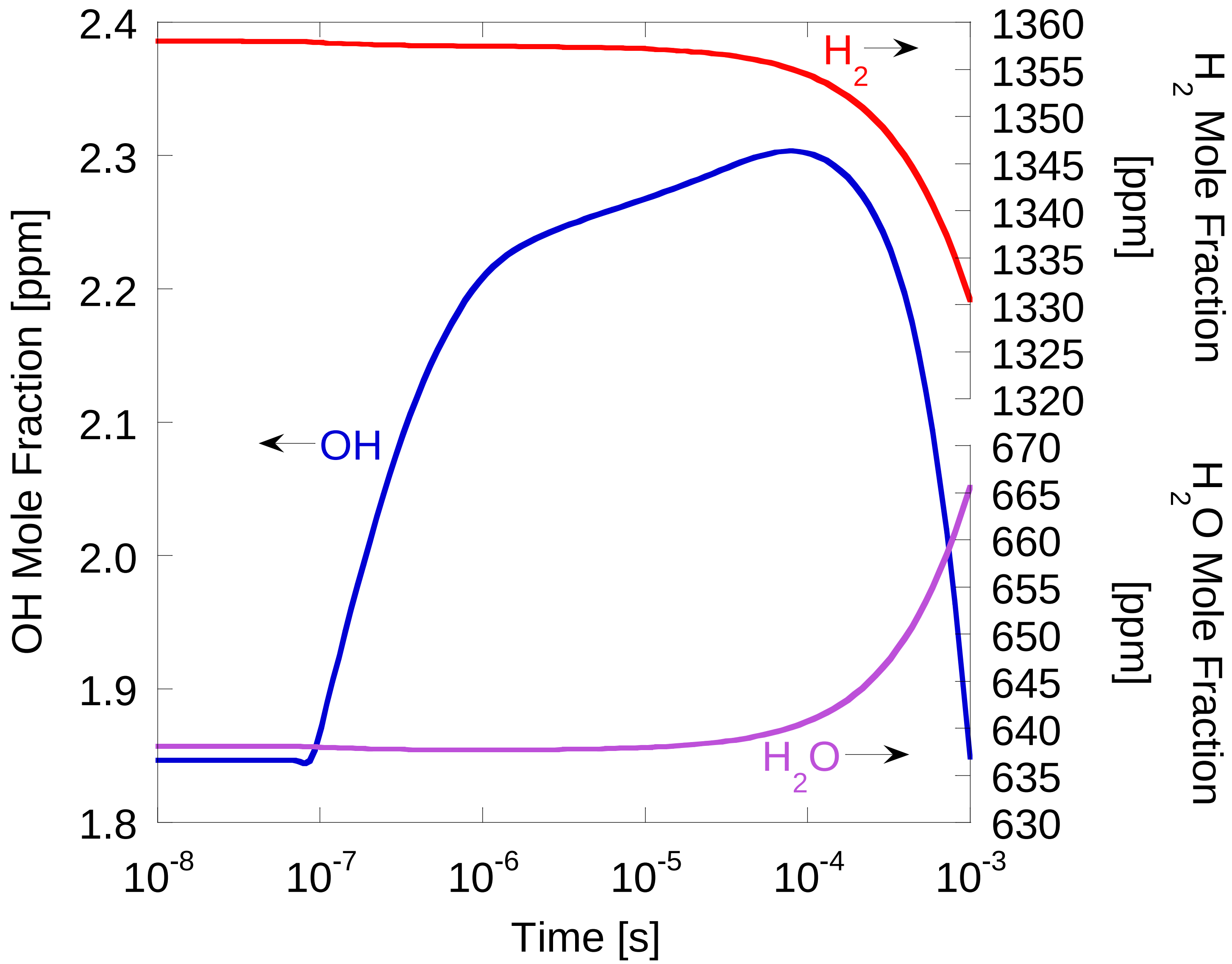
Details of Individual Pulse

Conditions: P = 1 atm, T=800 K, Q = 1 LPM, 2000ppm H2/3000ppm O2 / Ar by balance

Plasma Conditions: 10kV, v = 1 kHz

Pulse #22

Discharge at 77 ns, V_{peak} at 100 ns



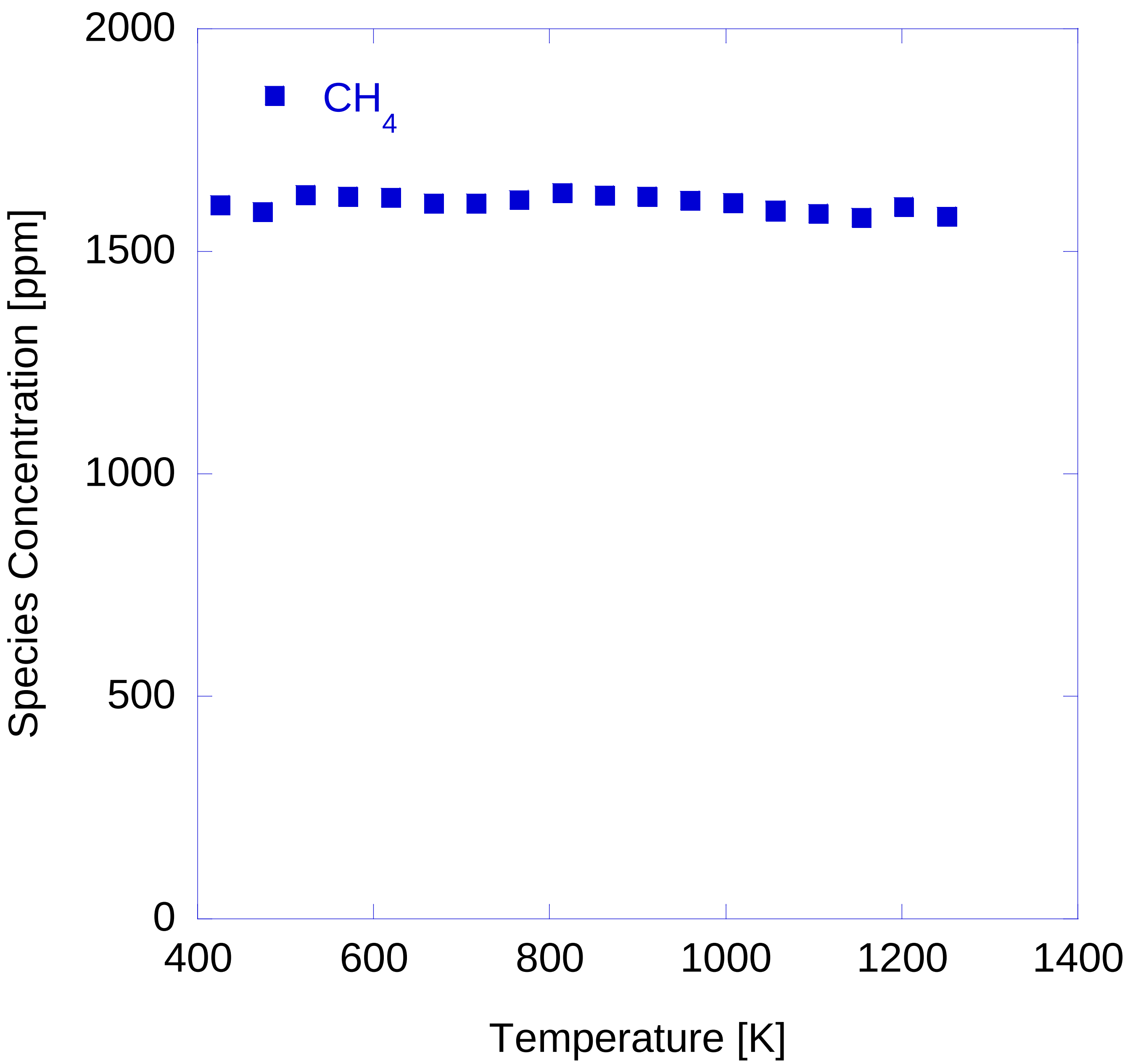
Raw Data for Hydrocarbon Experiments

Fourth Annual Review Meeting of the **AFOSR MURI** "***Fundamental Mechanisms, Predictive Modeling, and Novel Aerospace Applications of Plasma Assisted Combustion***"

22-24th October 2013, Basic Research Innovation Collaboration Center, Arlington, VA

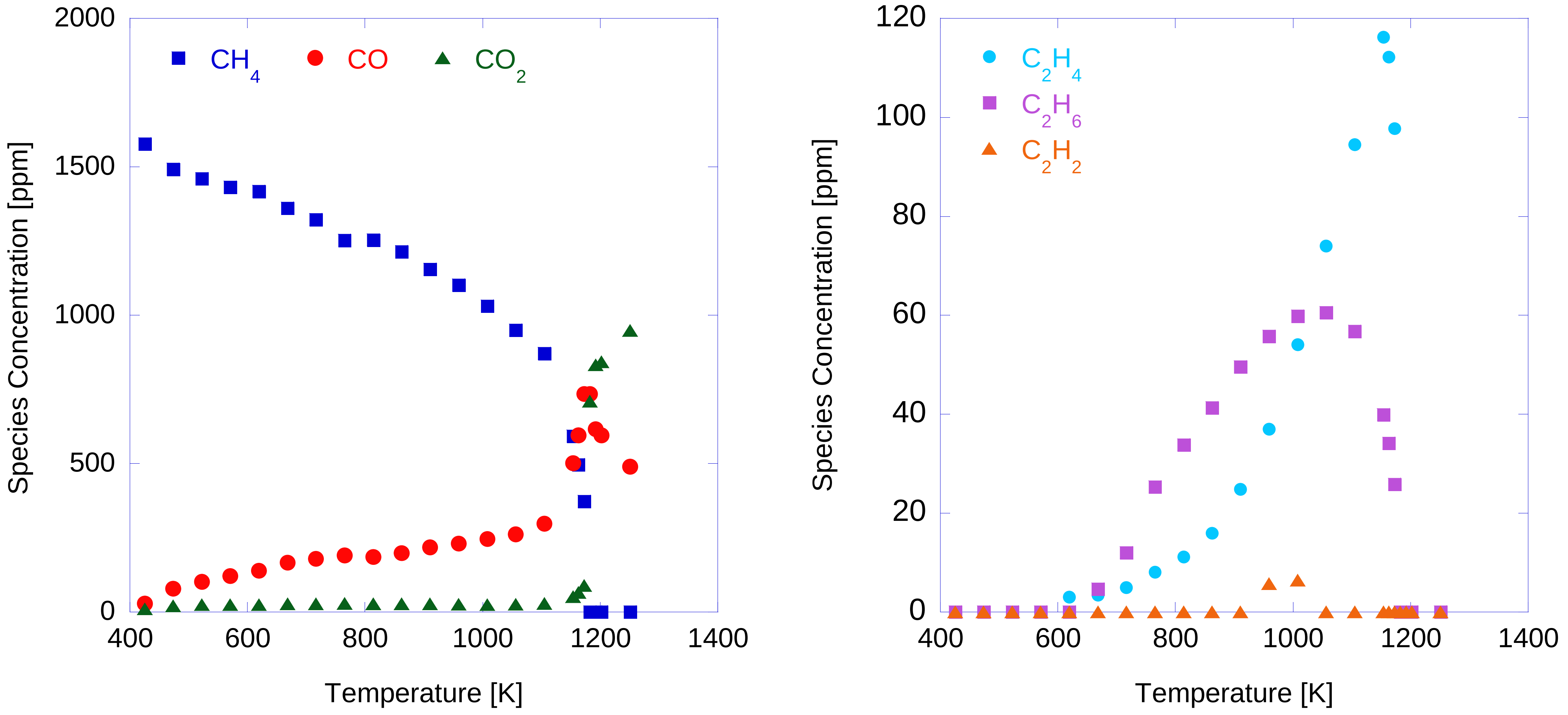
CH_4 Thermal Oxidation

Experimental Conditions: P = 1 atm, Q = 1 LPM, 1600ppm CH4/3000ppm O2 / Ar by balance



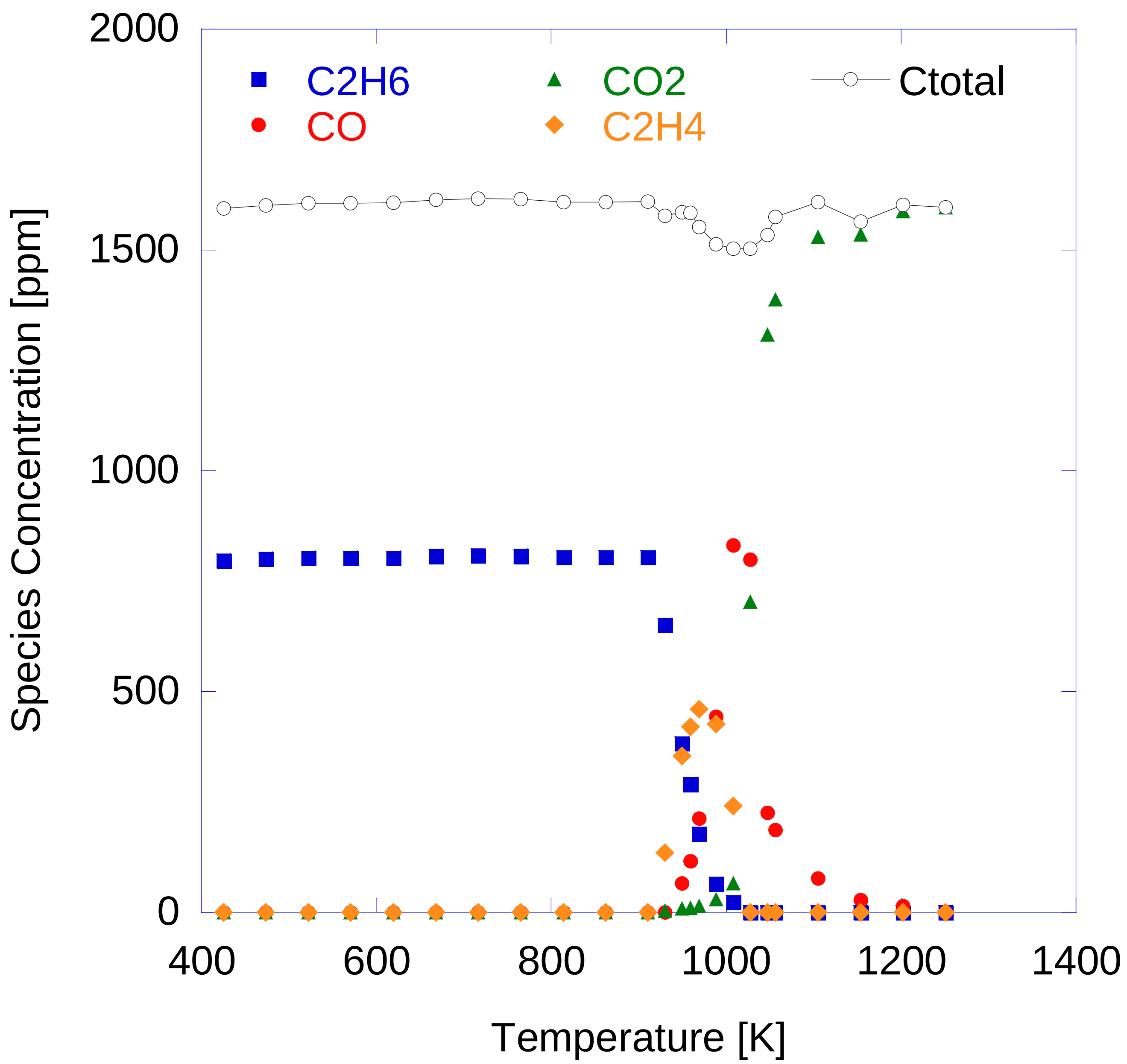
CH_4 Plasma Oxidation

Experimental Conditions: P = 1 atm, Q = 1 LPM, 1600ppm CH4/3000ppm O2 / Ar by balance
Plasma Conditions: 10 kV, *v* = 1 kHz

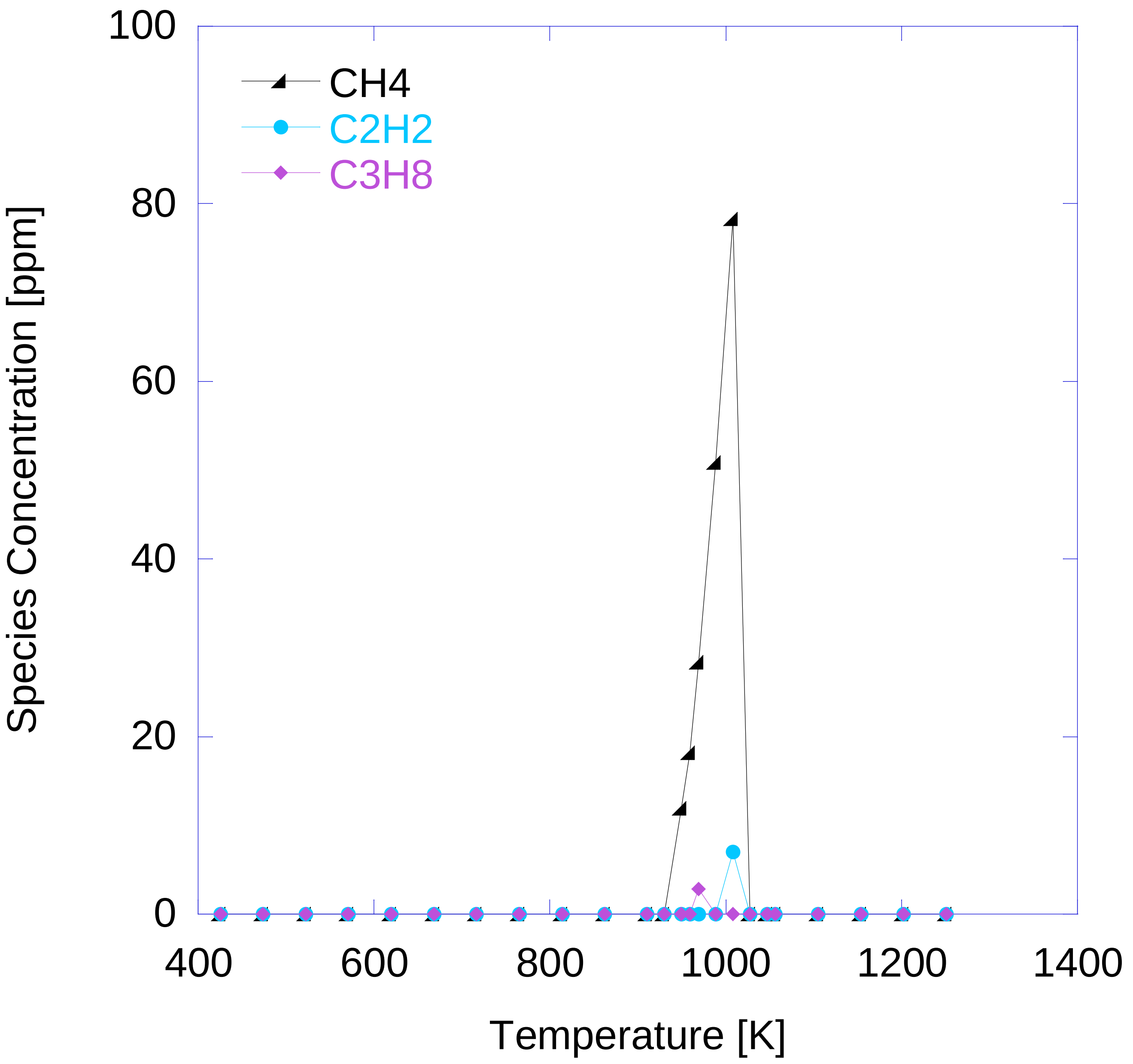


C_2H_6 Thermal Oxidation

Experimental Conditions: P = 1 atm, Q = 1 LPM, 800ppm C2H6/3000ppm O2 / Ar by balance



Major Species

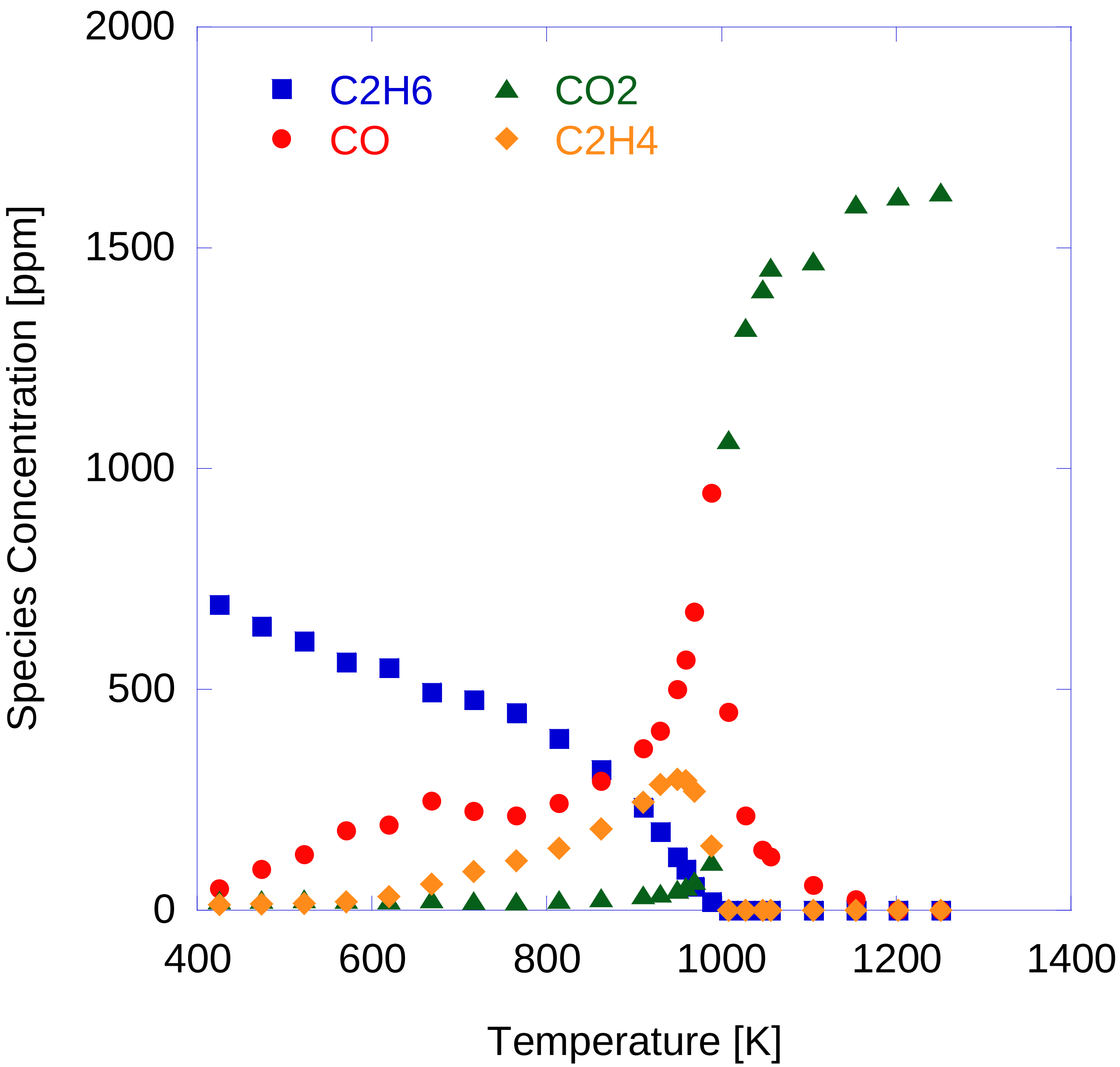


Minor Species

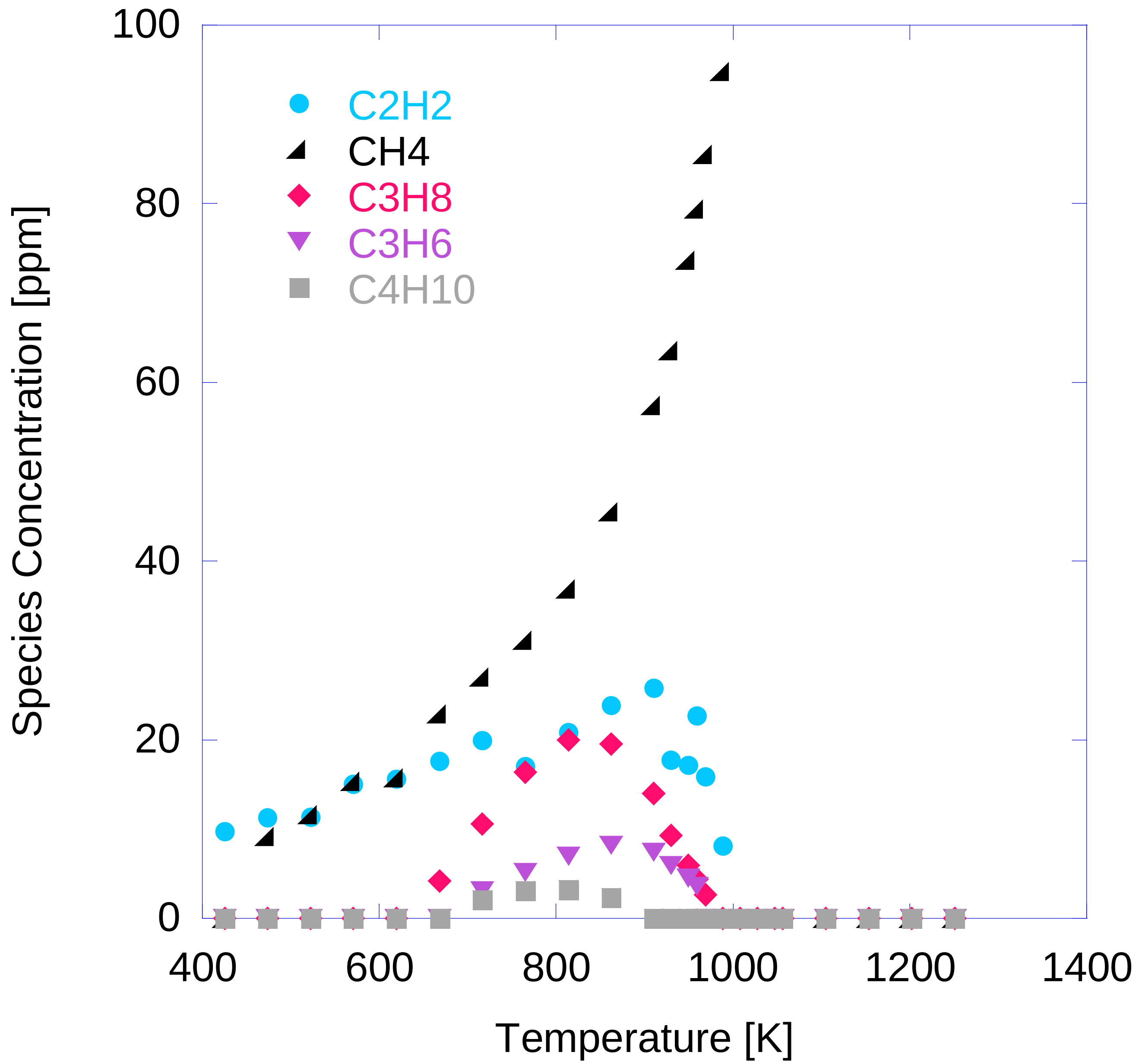
C_2H_6 Plasma Oxidation

Experimental Conditions: P = 1 atm, Q = 1 LPM, 800ppm C2H6/3000ppm O2 / Ar by balance

Plasma Conditions: 10 kV, v = 1 kHz



Major Species

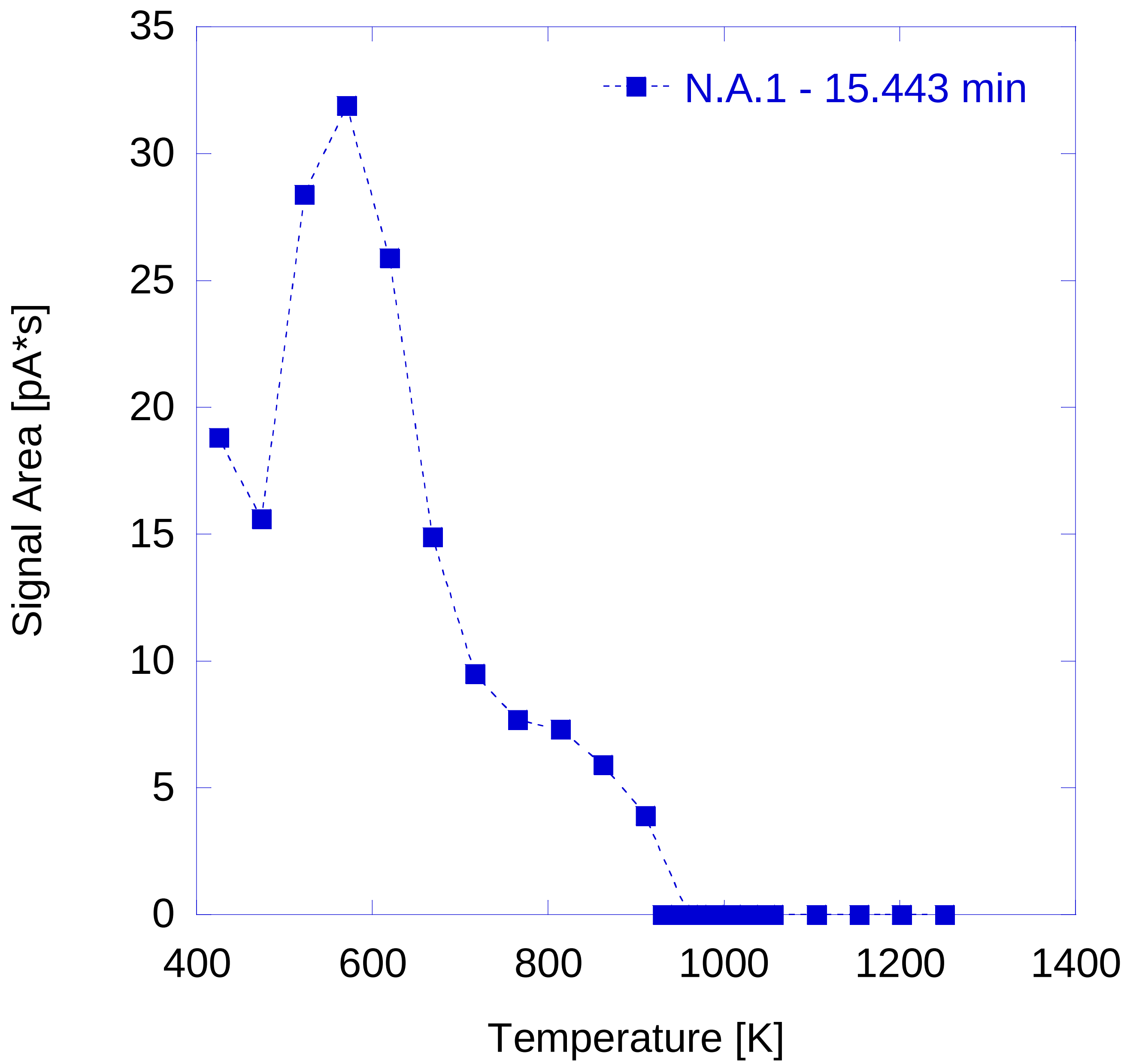


Minor Species

C_2H_6 Plasma Oxidation

Experimental Conditions: P = 1 atm, Q = 1 LPM, 800ppm C2H6/3000ppm O2 / Ar by balance

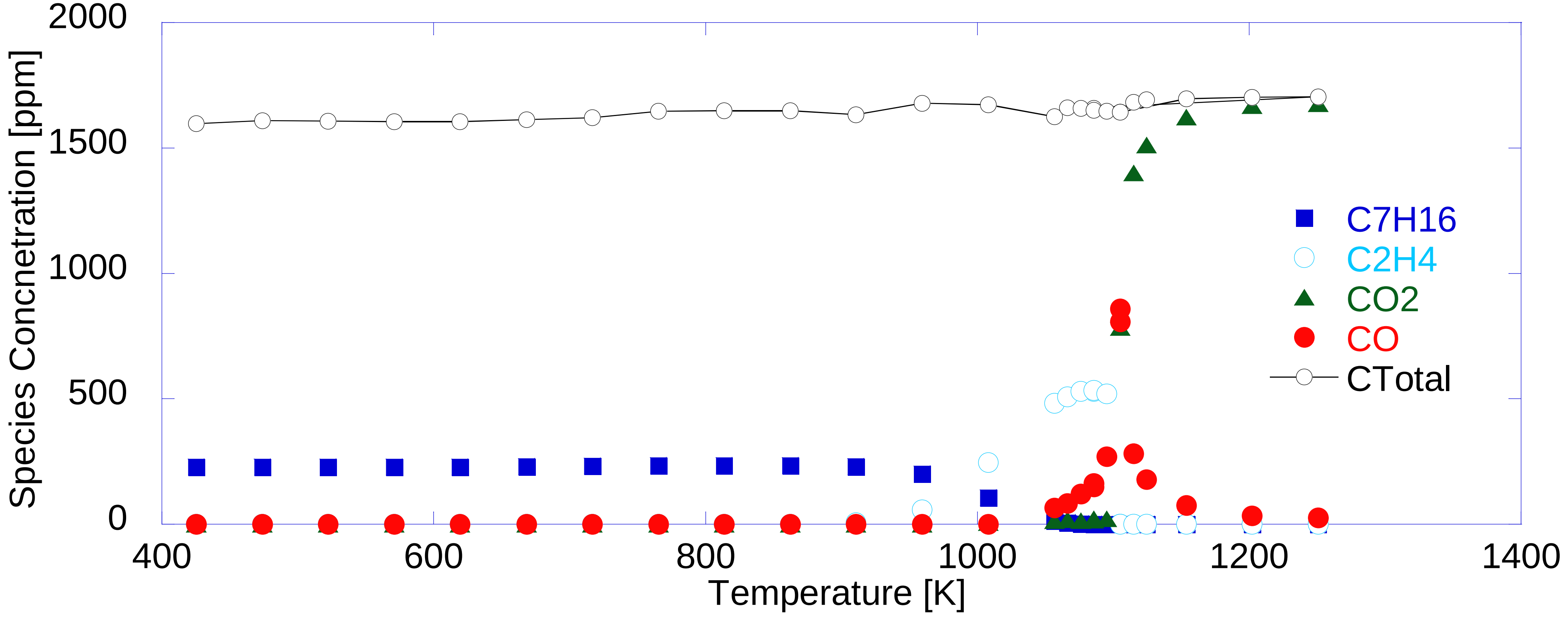
Plasma Conditions: 10 kV, *ν* = 1 kHz



Unknown Species

C_7H_{16} Thermal Oxidation – Major Species

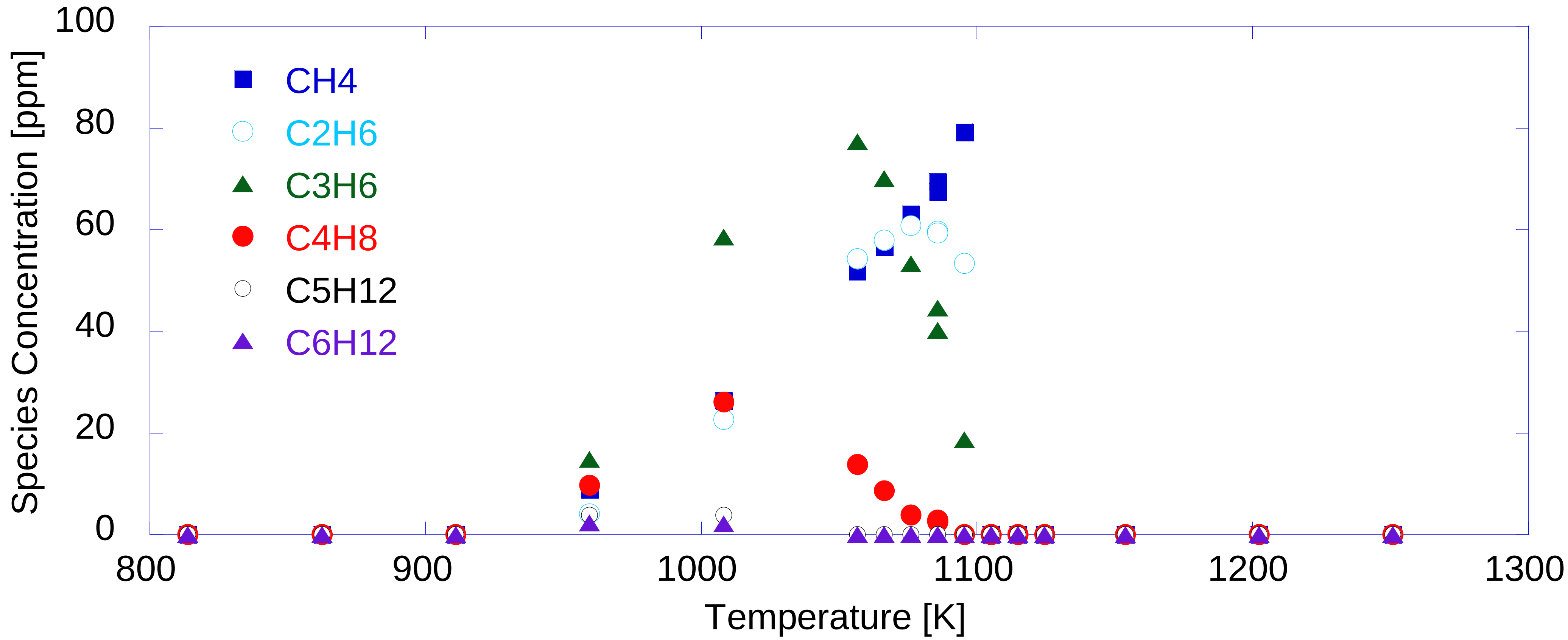
Experimental Conditions: P = 1 atm, Q = 1 LPM, 228ppm C4H10/3000ppm O2 / Ar by balance

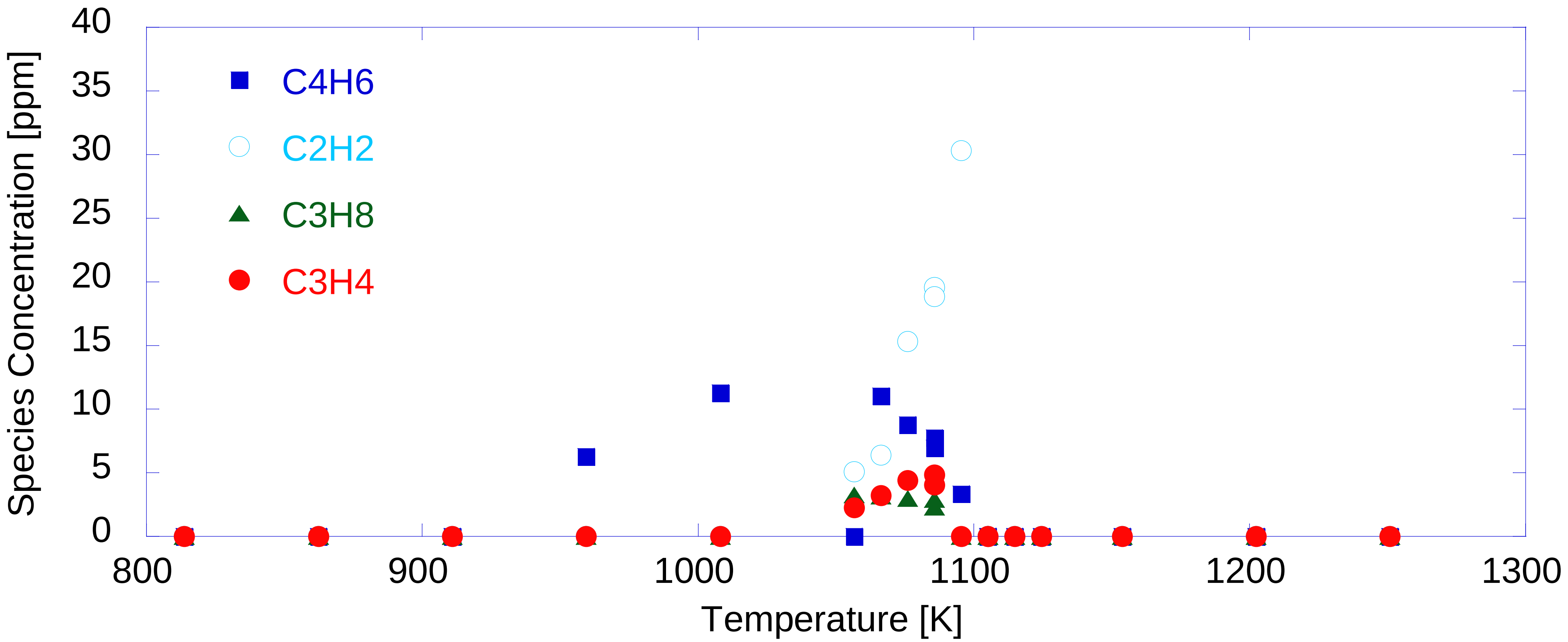


Major Species

C_7H_{16} Thermal Oxidation – Minor Species

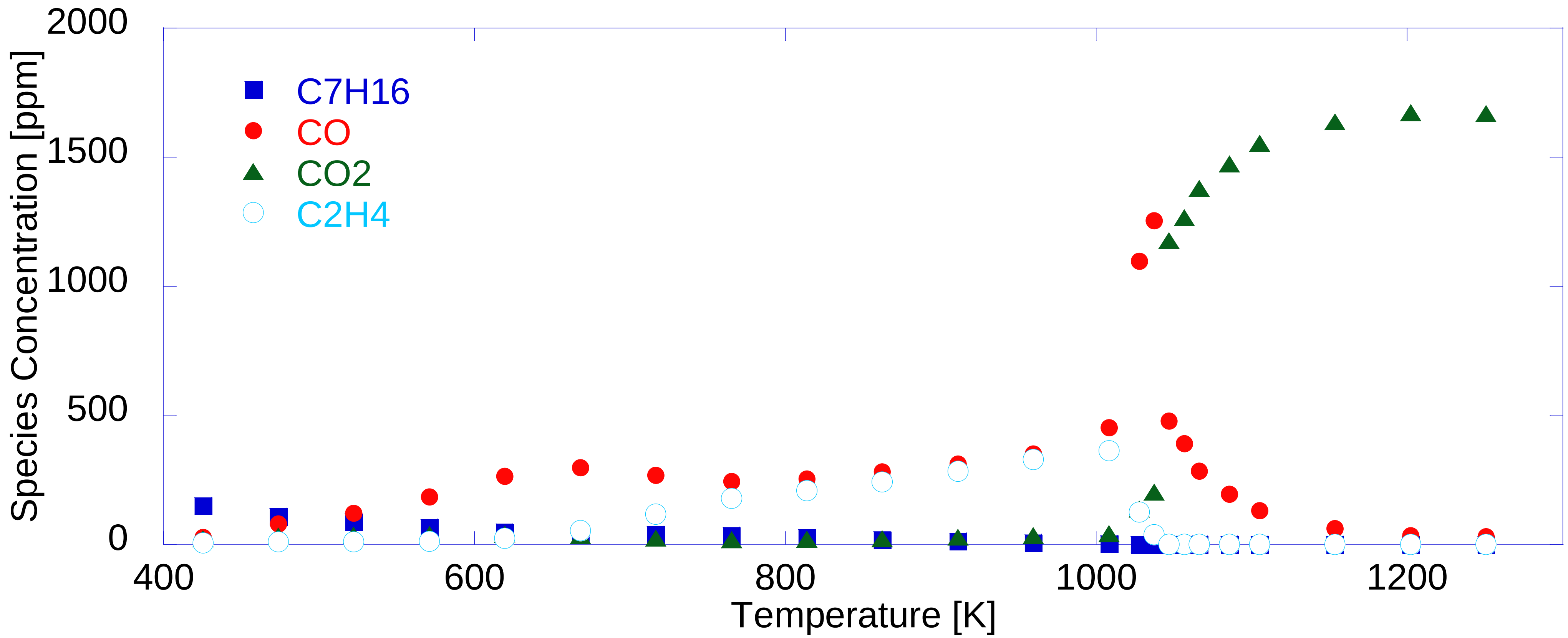
Experimental Conditions: P = 1 atm, Q = 1 LPM, 228ppm C4H10/3000ppm O2 / Ar by balance





C_7H_{16} Plasma Oxidation – Major Species

Experimental Conditions: P = 1 atm, Q = 1 LPM, 228ppm C4H10/3000ppm O2 / Ar by balance
Plasma Conditions: 10 kV, ν = 1 kHz

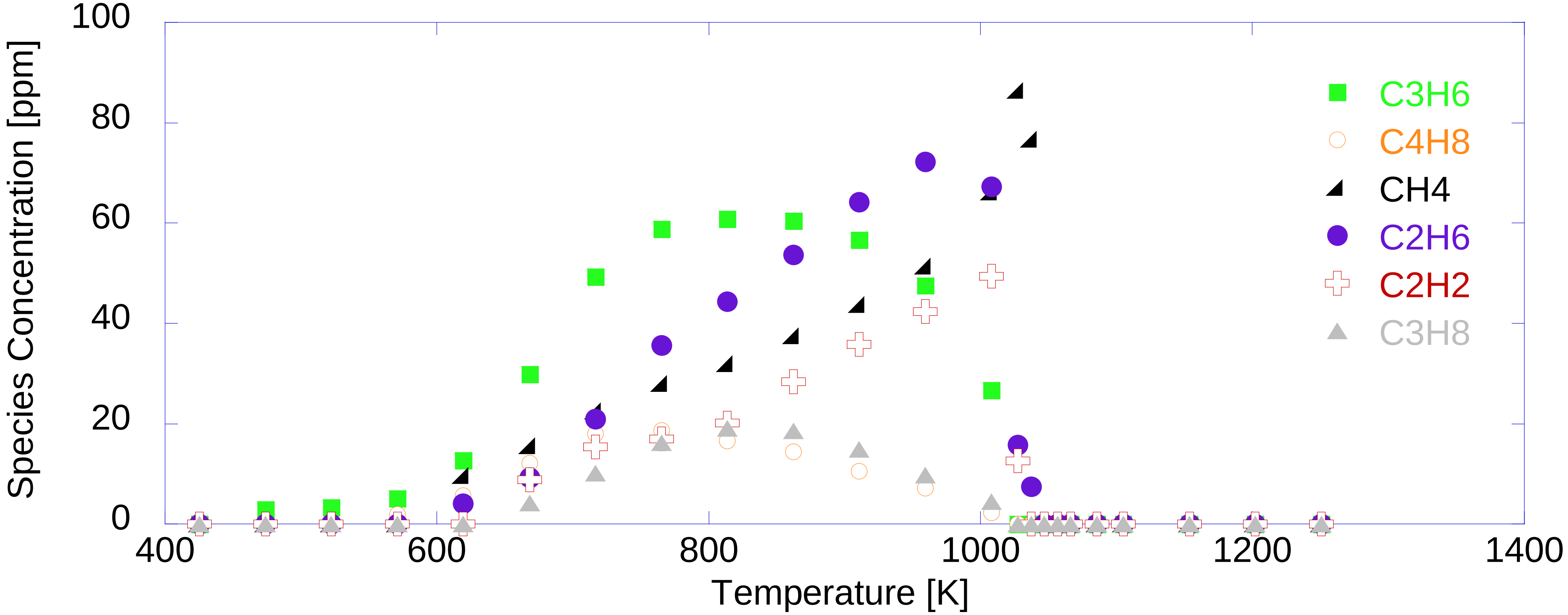


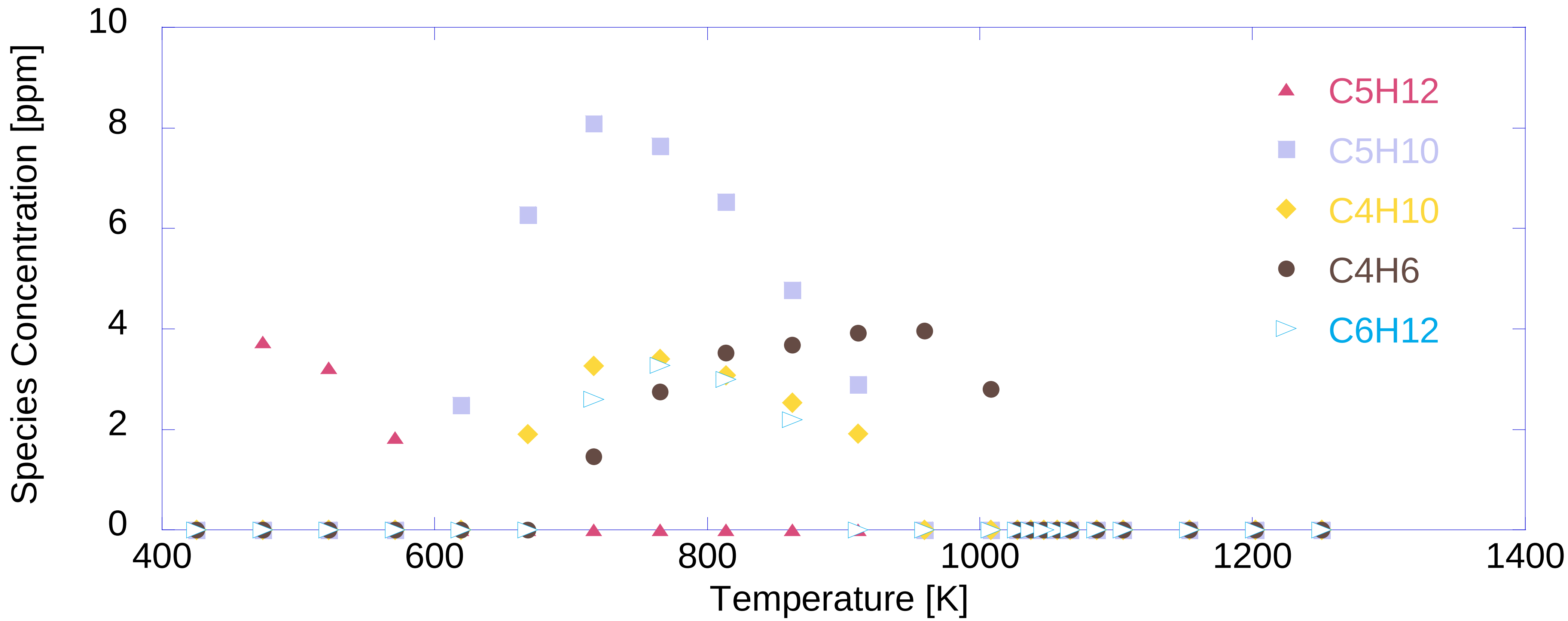
Major Species

C_7H_{16} Plasma Oxidation – Minor Species

Experimental Conditions: P = 1 atm, Q = 1 LPM, 228ppm C4H10/3000ppm O2 / Ar by balance

Plasma Conditions: 10 kV, ν = 1 kHz





C_7H_{16} Plasma Oxidation – Unknowns

Experimental Conditions: P = 1 atm, Q = 1 LPM, 228ppm C4H10/3000ppm O2 / Ar by balance

Plasma Conditions: 10 kV, ν = 1 kHz